Diamond Machining in 5 wt\% Y\textsubscript{2}O\textsubscript{3}  
Sinter Hipped Silicon Nitride

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ABSTRACT

A collaborative research project between Rolls-Royce Plc, Leavesden, the Physics Department, University of Surrey, and the former Central Electricity Generating Board, Gravesend, was set up to study peripheral diamond wheel machining damage in silicon nitride ceramics. The objective of the work to be carried out at the University of Surrey was to study the nature and depth of machining damage in 3 point flexural rupture test bars made from 5 wt% Y₂O₃ sintered hot isostatically pressed silicon nitride. The bars were machined to three surface finishes. The work carried out at Rolls-Royce by Mr R Quinn concentrated on the effects the machining damage had on the fracture strengths of the test bars. At the CEGB, Mr P E J Flewitt carried out X-ray diffraction stress tests to analyse residual surface stresses caused by the diamond machining.

Work at the University of Surrey has identified in detail the nature and depth of machining damage in "coarse" 0.4μm centre line average roughness surfaces. Deep grooves up to 2μm depth, 18μm width are found to have been superimposed on the general surface roughness by singularly large diamonds in the 350 grit diamond wheel. Sub-surface median cracks normal to the machining direction were clearly identified in bar cross-sections using oblique, diffuse "penumbra" illumination in an optical microscope, an as yet undocumented technique. Cross-section views of the machined surface and sub-surface were made possible by the very difficult and delicate technique of producing sandwich cross-sections of the machined bars. An analysis of machining-induced median cracks has not been carried out in such detail before. Semi-elliptical in shape the median cracks extend from 6μm - 45μm below the machined surface, and range from 19μm to 101μm in length parallel to the machining direction. They initiate at the focal point of a tributary system of microcracks at an average depth of 4μm - 5μm below the machined surface. It is believed that the median cracks initiate at the plastic/elastic boundary of a plastically deformed surface layer. Therefore a residual compressive layer, formed by the overlap of localised residual stresses from multi-particle contact events,
and bound by an underlying tensile field, is thought to have an average depth of 4μm - 5μm. A very innovative technique was used to reveal sub-surface deformation, where TEM X-ray microdiffraction spots were distorted by mechanical damage in the ceramic grain structure. The "arcing" or "streaking" of the diffraction spots tended to disappear at a depth of 4μm - 5μm below the machined surface. This is further evidence of the existence of a thin layer in residual compression, which has an average depth of 4μm - 5μm. This technique is not known to have been used before.

Fine diamond machining with a 600 grit wheel produced a centre line average roughness of 0.01/0.02 μm. However, evidence of machining damage is still present in the form of "remnant tracks" which lie parallel to the machining direction and consist of material pull-out. They are remnants of machining damage under grinding grooves introduced in previous machining stages.

Single point Vickers pyramid diamond scratches were implemented at different loads on a polished surface. The morphology of the grooves and material fragmentation and the sub-surface median cracks were examined. Many features were found to resemble the deformation/fracture formed under a deep grinding groove in the coarse machined surface.

Work carried out at Rolls-Royce by R Quinn showed that an increase in the quality of surface finish is accompanied by an increase in the mean strength and Weibull modulus of the machined bars. Furthermore a distinct anisotropy in the fracture strengths parallel and normal to the "coarse" machining direction is evidence of anisotropy in machining damage formed by a peripheral diamond grinding wheel. X-ray diffraction tests carried out at the CEGB by P E J Flewitt showed that machining damage produces a long range biaxial residual compressive field with the highest component acting normal to the machining direction. These results are consistent with the nature of machining damage identified at the University of Surrey, namely the strength-controlling median cracks which lie parallel to the machining direction and the residual compressive stress which exists as a thin 4μm - 5μm layer below the machined surface.
Processing flaws were discovered in the as-hipped billets received for the project. Their elemental composition and likely origin were examined. A three dimensional "cellular network" flaw ranging from 400μm to 2.1mm in size (in different production batches) is believed to have been formed as a result of flocculation clustering during processing. Clusters of 1μm - 3μm metallic particles were also identified. They range from 5μm - 45μm in size. The contaminant particles are steel and were introduced as a result of the original ceramic powder ball milling process which employed a steel ball mill.
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Electron microscopy has been a major tool in the study of machining damage and the Micro Structural Studies Unit at the University of Surrey has provided all top grade technical facilities and advice required by a materials scientist. Over the years I have worked with various microscope operators, some who have moved on to work at different locations and establishments. I would like to thank Mark Smithers, Gill Gibbs, Stuart Godfrey, Mark Dallas, and Vernon Watkins who helped me with the delicate final stages of ion beam thinning sandwich cross-sectional specimens, and the subsequent difficult transmission electron microscope analysis. Dawn Chescoe has provided an excellent overall management of this unit and has offered good practical advice throughout.

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NOTES
Tables and figures will be found in the text close to the relevant section.
CHAPTER 1. INTRODUCTION

Over the last 20 years considerable research has been carried out on high performance materials which could assume the role of metals in high temperature engineering applications. In the aerospace and automotive industries there has been a growing awareness of the need to improve energy conservation, using materials which will undergo more severe service conditions involving higher temperatures and mechanical stresses, and a corrosive environment. With improved performance and reduced cost of ownership as the major objectives advanced silicon nitride ceramics have been leading contenders for application in (aero) gas turbine and reciprocating heat engines due to their exceptional properties: low density (half as dense as steel), high strength and wear resistance, stable up to 1800°C, chemical stability, resistance to creep, low coefficient of thermal expansion and resistance to thermal shock. The properties of a range of ceramic materials are presented in Table 2.2, Section 2.3.4. However, 20 years of private and government funded research and development by the major industrial nations (USA, Japan, Germany, Italy, France and the UK) have failed to produce a fully developed commercial ceramic engine. One of the major reasons for this is the difficulty in fabricating fully dense, precisely shaped components with predictable and reliable properties. This difficulty results from the fact that ceramic materials, different to metals, are hard and brittle in nature and fracture occurs from discrete flaws which are activated by an applied or concentrated stress. The flaws that cause this intrinsic brittleness are created either during the fabrication route or by contact damage after fabrication. Thus ceramics are far less tolerant to machining or contact damage than metals. The minimisation of flaws and therefore the reduction of their effect on (cold) fracture strength is important if reliable components are to be made from brittle materials for load bearing applications.

A collaborative research project between Rolls-Royce Plc, Leavesden, the Physics Department, University of Surrey, and the former Central Electricity Generating Board (CEGB), Gravesend, was set up to study machining damage in silicon nitride ceramics. The original objective of the work to be carried out at the
University of Surrey was solely to study the effects in the microstructure of diamond machining 5 wt% Y$_2$O$_3$ sinter hipped silicon nitride ceramic: in essence to study the nature and depth of machining damage in the surface and sub-surface of 3 point flexural rupture test bars. The work carried out at Rolls-Royce by Mr R Quinn concentrated on the effects the machining damage had on the fracture strengths of the test bars. At the CEGB, Mr P E J Flewitt carried out X-ray diffraction stress tests in order to analyse the surface and sub-surface residual stresses caused by the diamond machining.

However, the machined specimens were not received until after one and a half years into the project due to unavoidable delays in materials processing. Therefore in the meantime a programme of work was carried out on experimenting and refining specimen preparation techniques on a variety of available silicon nitride ceramics. In the study of mechanical damage caused by diamond machining, specimen preparation is a very important process. One has to be completely sure that the observed damage is due to real artefacts of the machining process, and not damage caused by one or more of the numerous specimen preparation stages. Preparation processes may not only produce separate defects, but these may subsequently interact with the existing machining damage, thus giving a false interpretation of the effects of diamond machining. It is therefore imperative that each stage of specimen preparation is carried out in such a way that these problems do not occur, and with brittle ceramics this is a very delicate and difficult task. Extensive work was carried out to research and refine each preparation stage so that a carefully controlled process would produce quality defect-free specimens for examination. The short summary of the work is set out in Appendix B and should be an invaluable assistance to researchers who are either experienced or working on ceramics for the first time. There will be a number of references to Appendix B throughout the thesis.

After eight months of the project a complete as-hipped 5 wt% Y$_2$O$_3$ sintered silicon nitride (HIP) billet was supplied. This was fabricated by Turner and Newall Ltd and finished in an August 1987 HIP run at ASEA Cerama AB, Sweden. It was immediately apparent that the material contained various impurities, and
these were examined and characterised. The machined specimens were ultimately cut from a new batch of material fabricated in an April 1988 HIP run at ASEA. Due to the urgency in directly studying the effects of diamond machining, when the specimens finally arrived, a very short analysis was made of the existent processing defects.

Thus Part A of the thesis briefly covers the investigation and determination of the elemental composition and likely origin of processing flaws existent in 5 wt% Y₂O₃ sinter hipped silicon nitride ceramic.

In Part B, the main part of the thesis, the effects of diamond machining in silicon nitride ceramic are investigated. Residual stresses left by grinding material surfaces has been the subject of considerable study this century. Recently, with the arrival of advanced technical ceramics, their importance has increased, and their influence on the mechanical properties of a material is an area of intense interest. In order to achieve the fine tolerances and quality surface finishes required from a structural engineering ceramic component, some form of machining is often necessary; the state-of-the-art forming processes do not have an adequate precision capability and they may leave a material with surface processing flaws and reaction layers. The strength of a component is strongly dependent on the integrity of the surface and sub-surface layers, and therefore on the flaw size and distribution. Thus the objective of this work was to study the macroscopic and microscopic features in machining damage caused by diamond grinding to different surface finishes, and to provide an estimation of the depth of damage in the surface. There are various machining techniques available for ceramics, the most common involving a rotation grinding wheel where the workpiece traverses under the wheel surface. This method was used on three point flexural rupture test bars where the peripheral grinding wheel was set to traverse parallel, and normal, to the length of the bars. This would allow an analysis of any directionality in the effects of machining damage on i) the microstructure (carried out at the University of Surrey), and ii) the fracture strengths (carried out at Rolls-Royce). Three surface finishes were selected to be tested, nominally of 0.4μm, 0.1/0.2μm and 0.05μm centre line average roughness, and in the
collaborative project these were known as "coarse, intermediate and fine" surface finishes respectively.

The presence of residual stresses has already been detected by various workers via different methods, encompassing indentation flaw effects, X-ray diffraction techniques, and bar curvature measurements (Samuel and Chandrasekar 1989). These methods have described the nature of residual stresses and related the magnitude of the stresses to the particular grinding conditions. However, no attempts have been made to define and directly measure the depth of the residual compressive layer, other than estimates deriving from indirect measurements or interpolations.

At the University of Surrey, using a cross-sectional technique, the machined surface and sub-surface layers were investigated normal and parallel to the machining direction to determine the nature and depth of damage (therefore the subsequent residual compressive layer), and also the extent of sub-surface crack formation. The former was carried out by using a cross-sectional TEM technique whereby the extent and depth of deformation was detected with X-ray microdiffraction analysis, and the latter used an optical microscope technique as yet undocumented, where cracks were highlighted with "penumbra", diffuse, oblique illumination. A description of these two damage parameters would allow an analysis of their role in the micromechanics of failure, and also a tentative prediction of fracture strengths. Some quantitative and qualitative comparisons could then be made with experimental measurements from flexural rupture strength tests carried out by R Quinn, (Rolls-Royce PLC, Leavesden) and X-ray diffraction stress tests by P E J Flewitt, (CEGB, Gravesend).

The model used in this thesis to describe the effects of machining damage begins with the analysis of a single elastic/plastic contact event, (ie an indentation formed by a Vickers pyramid indentor), and the subsequent residual stress and fracture. By using this simplified example the effect of grinding a surface can be understood by considering the result of many particle contact events and the overlapping of residual fields. This model predicts the existence of a residual compressive layer with an underlying compensating tensile component, and local tensile stresses acting under strength-controlling flaws. A quantitative description of the effects of
applying a tensile stress on a surface containing a single indentation is understood. However, the analysis of ground surfaces has proven to be elusive as the role played by the residual stresses and strength-controlling flaws is very complex and as yet not completely understood. The proposed model, therefore, can provide a tentative estimate of the fracture strengths derived from the machining damage parameters.

The thesis is essentially divided into two parts, eight chapters and two appendices as follows:

Chapter 1. Introduction
Chapter 2. Fabrication of Silicon Nitride Ceramics

Part A
Chapter 3. Elemental Analysis of Processing Flaws in 5 wt% Y_2O_3 Sinter Hipped Silicon Nitride

Part B
Diamond Machining in 5 wt% Y_2O_3 Sinter Hipped Silicon Nitride

Chapter 4. Theory
Chapter 5. Experimental Procedure
Chapter 6. Experimental Results
Chapter 7. Discussion
Chapter 8. Conclusions
Appendix A. Fabrication and Applications of Silicon Nitride Ceramics
Appendix B. Specimen Preparation

To give the reader an at-a-glance idea of the contents of the thesis, short summaries are provided before each chapter. In Chapter 7 the results from the study of machining damage are discussed, and are also tied in with the results from fracture strength tests carried out at Rolls-Royce by R Quinn, and X-ray diffraction residual stress measurements carried out at the GECB by P E J Flewitt. Chapter 8 provides short sentences which concisely highlight the main findings of the whole collaborative research project.
CHAPTER 2. FABRICATION OF SILICON NITRIDE CERAMICS

SUMMARY

This chapter introduces the main methods of forming silicon nitride ceramics and describes in particular the fabrication and applications of sintered hot isostatically pressed silicon nitride.

2.1 INTRODUCTION

The principal forming techniques of silicon nitride ceramics are introduced, together with an explanation of how the control of fabrication conditions produces different types of ceramic, each with their own mechanical and thermal properties.

2.2 CRYSTAL CHEMISTRY

The general crystal chemistry of the silicon nitride structure is detailed on an atomic level.

2.3 SINTERED HOT ISOSTATICALLY PRESSED SILICON NITRIDE

An introduction describes how the performance of a material is dependent on the microstructure, which can be controlled by the ceramic constituents and fabrication conditions. The fabrication of sintered silicon nitride is then detailed, starting from the initial powder through to the sintering process, and this is accompanied by a description of the chemical reactions and final microstructure formed. A comparison with the properties of different types of silicon nitride, other engineering ceramics and metals is given.

A potentially major application for ceramics is in aero gas turbine engines, principally the compressor, combustion chamber and the turbine. The mechanical and thermal conditions these components have to endure are described, as well as how sintered silicon nitride might provide an improvement or solution to current materials limitations.
CHAPTER 2. FABRICATION OF SILICON NITRIDE CERAMICS

2.1 INTRODUCTION

The desirable properties of a silicon nitride component are produced in highly densified material. However, in practice this is difficult to achieve; the strong atomic covalent bonding in pure silicon nitride results in low self-diffusivity and the 1878°C temperature at which atoms begin to migrate is so high that the silicon nitride decomposes by volatilisation of nitrogen (Singhal 1976). Therefore a component cannot be fabricated to high density merely by firing, as is the case for silicate ceramics.

Reaction bonding with nitrogen gas facilitates densification, but materials of only 75-82% density are formed (See Appendix A). Sintering with densifying additives is a process that provides appropriate conditions for densification (Deeley et al 1961, Tsuge and Nishida 1978) and manipulation of the chemical compositions and process conditions produces a number of silicon nitride ceramics with a variety of microstructures and mechanical properties. Primary shaping is carried out by isostatic pressing, extrusion, injection moulding or slip casting. Complex shaped components such as turbine blades or rotors are formed by injection moulding. Secondary shaping is achieved by green machining. A brief discussion of a few ceramic types is given in Appendix A, and a description of 5 wt% Y2O3 sinter hipped silicon nitride as supplied by Turner and Newall Ltd for this project is given in Section 2.3 below.

2.2 CRYSTAL CHEMISTRY

Pure silicon nitride exists in two polymorphic forms as a covalently bonded 3-d superlattice structure of silicon nitride (Si3N4) tetrahedra, where each nitrogen atom is common to 3 tetrahedra (Figure 2.1). The β phase has a density of 3.190 gm cm⁻³ and contains an hexagonal unit cell (Si₆N₆), with cell dimensions a₀ = 7.608 Å and c₀ = 2.911 Å. The α polymorph also contains an hexagonal unit cell (Si₁₂N₁₆) with roughly double the cell volume. The cell dimensions are a₀ = 7.752 Å and c₀ = 5.620 Å (c₀ ≈ 2 x c₀), and the density is 3.167 gm cm⁻³.
BOND LENGTHS

Si - N1 1.730
Si - N1 1.739
Si - N2 1.745

Figure 2.1 The crystal structure of $\beta$-Si$_3$N$_4$
2.3 SINTERED HOT ISOSTATICALLY PRESSED SILICON NITRIDE

2.3.1 Introduction

The fabrication of highly dense, complex shaped components requires the use of hot pressing conditions. Uniaxial hot pressing is avoided for particular applications, however, as an aligned microstructure parallel to the pressing direction is formed. Hot isostatic pressing overcomes this problem and only a minimum amount of sintering additives are required to achieve a near theoretical density. A cold isostatic pressed starting powder compact plus a metal oxide sintering additive (or a previously reaction bonded silicon nitride component) is encapsulated and heated to high temperature, and it is the high applied pressure that reduces the requirement of sintering additive, as compared to pressureless sintered material. The volume and composition of sintering additives is very important as the mechanical properties of a ceramic material at high temperatures is strongly dependant on the integrity of the intergranular phase. A glassy intergranular phase tends to soften at high temperatures (1000°C for MgO in HPSN), causing degradation of the material via grain boundary sliding and low creep resistance. Control of the liquid-phase sintering process allows the formation of materials with different properties, and a compromise is reached depending on the final component operating conditions. A high glass fraction liquid sintering medium produces a higher $\beta$-$\text{Si}_3\text{N}_4$ grain aspect ratio, but the microstructure tends to be more anisotropic. A low glass fraction sintering medium is less viscous, thus producing less anisotropy, but the aspect ratio is lower. An improvement in the material creep resistance can also be achieved by forming higher temperature, more oxidation resistant intergranular glassy phases, and in smaller volumes (Loehman 1979). However, complete crystallisation of the intergranular phase produces a material that can operate at temperatures up to 1400°C without severe degradation of the mechanical properties (Lee et al 1988).

2.3.2 Powder Billet Formation

The starting silicon nitride powder, supplied by UBE Industries Ltd, was a high purity UBE E10 grade. The analytical
specifications (Table 2.1) show an oxygen content of 1.3 wt%, and this was assumed to be related to the SiO$_2$ on the outer surface of the powder particles, this being equivalent to 1.93 wt% SiO$_2$ impurity content. The metal oxide sintering additive supplied by Ventron Alpha Ltd. was a 99.99% pure Y$_2$O$_3$ powder. At Turner and Newall Ltd a 5 wt% Y$_2$O$_3$ and 95 wt% silicon nitride powder combination was milled in plastic cylinders, using propanol as the medium. The milled mixtures were dried, sieved and cold isostatically pressed to form green stage billets, which were then machined to a diameter of 22mm.
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<td></td>
</tr>
<tr>
<td>DEGREE OF CRYSTALLINITY</td>
<td>~100%</td>
</tr>
<tr>
<td>BETA/ALPHA AND BETA</td>
<td>3.0%</td>
</tr>
</tbody>
</table>
2.3.3 Hipping Process

There are few large scale hipping facilities in Europe at the present time, and only ASEA Cerama in Sweden had a capability to fabricate aero engine components.

At ASEA the green stage billet compacts were coated with a thin boron nitride layer and encapsulated in a Pyrex glass ampoule (or glass powder), which is contained within a graphite crucible. The boron nitride is applied as it facilitates the removal of the billet after hipping.

As the hipping process at ASEA is a proprietary technique detailed information on the encapsulation conditions or the hipping cycle was not available. Therefore it is assumed that the billets were first heated to a temperature of approximately 650°C under a nitrogen pressure of 2 MPa, to anneal the Pyrex ampoule/powder encapsulant. Then the temperature was increased to a sintering condition of 1700 - 1750°C, with an applied pressure of 100 - 200 Mpa, and maintained for about one hour. After processing the cooling conditions would be carefully controlled to allow complete crystallisation of the intergranular phase.

2.3.4 Reaction and Microstructure

At the hot pressing temperature the Y\textsubscript{2}O\textsubscript{3} additive reacts with the SiO\textsubscript{2} present on the surface of each SiN particle to form a silicate liquid. This allows mass transportation and densification by liquid phase sintering. The α - SiN powder dissolves in the liquid and on cooling, a solution precipitation reaction produces β-SiN grains and a binary eutectic intergranular phase. Of four Y - Si - O - N polymorph phases, only two are stable; Yttrium disilicate, Y\textsubscript{2}O\textsubscript{3} (SiO\textsubscript{2})\textsubscript{2}, and Yttrium - nitrogen - apatite, Y\textsubscript{5}(SiO\textsubscript{4})\textsubscript{3}N (Figure 2.2). A near complete α to β - Si\textsubscript{3}N\textsubscript{4} transformation is achieved and a bimodal microstructure is formed (Figure 2.3), with large grains reaching 2μm diameter, 10μm length with the average matrix size being 0.1 - 1μm diameter and 1 - 5μm length. A complete α to β Si\textsubscript{3}N\textsubscript{4} conversion is important as the formation of localised pockets of acicular α silicon nitride can become sites for fracture nucleation. A bimodal microstructure helps to increase the fracture toughness of the material as the predominant mode of failure in silicon nitride is via intergranular fracture; fine
Figure 2.2 Si$_3$N$_4$ - 3 SiO$_2$ - 2Y$_2$O$_3$ - 4YN phase diagram in equivalent units at 1550°C from Phase Diagrams for Ceramicists 1983
Figure 2.3 Schematic diagram of the densification of a Si₃N₄ ceramic via an additional sintering liquid.
grained materials tend to exhibit higher fracture toughness than coarse grained ones, but a bimodal microstructure is a further improvement as an intergranular crack is forced to propagate via a more tortuous route, and therefore, has to expend more energy.

Typical properties of sinter hipped silicon nitride are presented in Table 2.2 together with properties of a range of ceramic materials. Density measurements were carried out at Turner and Newall Ltd, and were found to be 3.25gm cm\(^{-3}\), 99.9% theoretical density. Three point bend tests were carried out by R Quinn at Rolls-Royce PLC Leavesden, measurements of Young's modulus and Poisson's ratio by Dr J Rider at the University of Surrey were made by ultrasonic wave velocity determination, and hardness and fracture toughness measurements by Mr R Holm at the University of Surrey were made by indentation/fracture tests.
<table>
<thead>
<tr>
<th>Physical Properties</th>
<th>3wt% Y₂O₃ Sinter-Hipped Silicon Nitride</th>
<th>Hot Pressed Silicon Nitride</th>
<th>Sintered Silicon Nitride</th>
<th>Reaction Bonded Silicon Nitride</th>
<th>Sintered Silicon Carbonide</th>
<th>Reaction Bonded Silicon Carbide</th>
<th>99% Al₂O₃ Alumina</th>
<th>5wt% Y₂O₃ Zirconia</th>
<th>EN32 Steel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (gm/cm²)</td>
<td>3.25</td>
<td>3.20</td>
<td>3.25</td>
<td>2.4</td>
<td>3.16</td>
<td>3.10</td>
<td>3.98</td>
<td>6.05</td>
<td>7.37</td>
</tr>
<tr>
<td>Open Porosity (%)</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>25</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Grain size (μm) (av)</td>
<td>0.5-2</td>
<td>-</td>
<td>1-2</td>
<td>-</td>
<td>1-2</td>
<td>1-10</td>
<td>0.5</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Poisson's Ratio</td>
<td>0.268</td>
<td>-</td>
<td>0.24</td>
<td>0.27</td>
<td>0.17</td>
<td>0.24</td>
<td>0.27</td>
<td>0.3</td>
<td>0.27</td>
</tr>
</tbody>
</table>
### Table 2.2: Properties of Sinter Hipped Silicon Nitride and a Range of Ceramics

#### Mechanical Properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Sinter Hipped Silicon Nitride</th>
<th>Hot Pressed Silicon Nitride</th>
<th>Sintered Silicon Nitride</th>
<th>Reaction Bonded Silicon Nitride</th>
<th>Sintered Silicon Carbide</th>
<th>Reaction Bonded Silicon Carbide</th>
<th>99% Al₂O₃ Alumina</th>
<th>5wt% Y₂O₃ Zirconia</th>
<th>EN32 Steel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardness HV₀.05 (kg mm⁻²)</td>
<td>1800</td>
<td>1800</td>
<td>1500</td>
<td>1100</td>
<td>2800</td>
<td>3000</td>
<td>1600</td>
<td>1400</td>
<td>450-650HB</td>
</tr>
<tr>
<td>Young's Modulus</td>
<td>322</td>
<td>310</td>
<td>290</td>
<td>170</td>
<td>450</td>
<td>390</td>
<td>360</td>
<td>200</td>
<td>206</td>
</tr>
<tr>
<td>Fracture Toughness KIC 20°C (MPa m¹/²)</td>
<td>4</td>
<td>-</td>
<td>8</td>
<td>3</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>9</td>
<td>-</td>
</tr>
<tr>
<td>Flexural Strength (3pt bend, 20°C) (MPa)</td>
<td>920 Mean</td>
<td>800</td>
<td>650</td>
<td>190</td>
<td>410</td>
<td>400</td>
<td>400</td>
<td>1000</td>
<td>-</td>
</tr>
<tr>
<td>Flexural Strength (3pt bend, 1000°C) (MPa)</td>
<td>850-1,020</td>
<td>-</td>
<td>450</td>
<td>190</td>
<td>410</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>Degrades</td>
</tr>
<tr>
<td>Flexural Strength (4pt bend, 1350°C) (MPa)</td>
<td>600</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Tensile Strength (20°C) (MPa)</td>
<td>500</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Compressive Strength (20°C) (MPa)</td>
<td>-</td>
<td>&gt;3000</td>
<td>2000</td>
<td>550</td>
<td>2000</td>
<td>2000</td>
<td>2700</td>
<td>2000</td>
<td>-</td>
</tr>
</tbody>
</table>
### TABLE 2.2: PROPERTIES OF SINTER HIPPED SILICON NITRIDE AND A RANGE OF CERAMICS

#### Thermal Properties

<table>
<thead>
<tr>
<th></th>
<th>5wtZrO₂ Sinter Hipped Silicon Nitride</th>
<th>Hot Pressed Silicon Nitride</th>
<th>Sintered Silicon Nitride</th>
<th>Reaction Bonded Silicon Nitride</th>
<th>Sintered Silicon Carbonide</th>
<th>Reaction Bonded Silicon Carbide</th>
<th>99% Al₂O₃ Alumina</th>
<th>5wtZrO₂ Zirconia</th>
<th>EN32 Steel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal Expansion Coefficient (20-800°C) (×10⁻⁶°C⁻¹)</td>
<td>3.7</td>
<td>3.2</td>
<td>3.0</td>
<td>3.0</td>
<td>3.8</td>
<td>4.3</td>
<td>8.3</td>
<td>8.0</td>
<td>13.0</td>
</tr>
<tr>
<td>Thermal Conductivity (Wm⁻¹°C⁻¹)</td>
<td>33</td>
<td>18</td>
<td>25</td>
<td>16</td>
<td>100</td>
<td>150</td>
<td>26</td>
<td>1.9</td>
<td>50</td>
</tr>
<tr>
<td>Specific Heat Jkg⁻¹°C⁻¹</td>
<td>–</td>
<td>800</td>
<td>800</td>
<td>800</td>
<td>1000</td>
<td>1100</td>
<td>390</td>
<td>540</td>
<td>–</td>
</tr>
<tr>
<td>Maximum Temp. of Use (°C) (cont), (short term, no load)</td>
<td>–</td>
<td>–</td>
<td>1150</td>
<td>1150</td>
<td>1400</td>
<td>1350</td>
<td>–</td>
<td>1000</td>
<td>–</td>
</tr>
<tr>
<td>Thermal Shock Resistance (9T)(°C)</td>
<td>–</td>
<td>600</td>
<td>650</td>
<td>600</td>
<td>380</td>
<td>400</td>
<td>–</td>
<td>250</td>
<td>–</td>
</tr>
</tbody>
</table>

#### Electrical Properties

| Electrical Resistivity (20°C) (ohm m) | – | >10¹⁰ | 10¹¹ | 10¹⁰ | – | 10 | 10¹³ | 10¹⁰ | – |
2.3.5 Applications

With improved performance and reduced cost of ownership being the major objectives in the design of aero gas turbine (AGT) engines, thrust to weight ratio and minimum fuel consumption are of paramount importance. The response to these objectives can be seen in the decreasing use of metals in manufacture (Figure 2.4a). From the 1940s to the present day thrust to weight ratios have improved from 3:1 to 8:1, and there are hopes that this will be increased up to 20:1 in the near future once new materials and design technology (new lighter, more resistant materials, reduced number and size of components) are fully integrated and standard. The overall pressure ratio and turbine entry temperature of the engine determine the thermal efficiency and therefore fuel consumption. The increase in pressure ratios over the same time period from 4:1 to 23:1 has significantly improved fuel efficiency and power and 30:1 is considered an achievable future target. Turbine entry temperatures have increased from 1400°C to 1950°C and are expected to reach 2300°C in the near future, but reaching the stoichiometric burning temperature of the fuel at around 2800°C is still a far off prospect. Sinter hipped silicon nitride has the highest temperature and strength characteristics of the silicon nitride (Figure 2.4b) and silicon carbide family of engineering ceramics, and therefore is the leading contender for application at the hot end of the aero gas turbine engine, replacing nickel-based superalloys (Figures 2.5a, 2.5b). HIP Si$_3$Ni$_4$ components would allow an increase in the turbine entry temperature and reduce the demand for cooling air, which is compromising in terms of power consumption, engineering design and component manufacture. An example of an AGT engine is the Gem 60 turboshift helicopter engine (Figure 2.6).

A description of the operation of an AGT engine, accompanied by a schematic cross-section diagram (Figure 2.7a), is as follows.

**The Aero Gas Turbine Engine**

**The Compressor**

The compressor, situated at the front of the engine, performs two functions - it draws air into the engine and it compresses it (in some engines by up to 30 atmospheres) before delivering it into the combustion chamber.
Figure 2.4a Trends in jet engine usage

Figure 2.4b Variation of typical flexural strengths with temperature for a range of Silicon Nitride ceramics
Figure 2.5a Strength of ceramics and nickel based superalloy

Figure 2.5b Improvements in temperature capability
Rolls-Royce Gem 60 turboshift

Figure 2.6 A helicopter aero gas turbine engine
Figure 2.7a (Source: Rolls-Royce plc)

FUTURE MILITARY ENGINE – POTENTIAL APPLICATION OF CERAMIC AND METAL-MATRIX COMPOSITES

Figure 2.7b Thermal, mechanical and environmental factors affecting the AMED blade (Source: Rolls-Royce plc)

Figure 2.7c Typical principal stress contours—vertical and mid-cross section of a turbine blade (Source: Rolls-Royce plc)
Compressors may be centrifugal and/or axial, the latter consisting of a number of stages of alternate rotating and stationary aerofoil-section blades which force the air through a convergent annular duct.

Many modern engines have more than one compressor because a high degree of compression requires a large number of compressor rows or "stages". Each stage has an optimum speed for best efficiency - the smaller the blades the higher the speed. If all the stages are on the same shaft, only a few of them will be operating at their optimum speed - the majority will be running either too fast or too slow. This problem is overcome by dividing the compressor into two or three parts, each driven by its own turbine and each rotating at its optimum speed. By this means, compression ratios up to 30:1 can be achieved, resulting in extremely high efficiency and very low specific fuel consumption.

The Combustion Chamber

The air from the compressor section, at anything up to 3 MPa, passes into the combustion chamber. This is an annular Ni-based superalloy "flame tube" or ring of tubes designed to achieve the most efficient combustion of the fuel/air mixture so that the maximum possible heat energy is extracted from the fuel in order to give the greatest rise in temperature and hence expansion of the gas.

The combustion chamber has a number of burners to vaporise the fuel before mixing it with the compressed air. Igniters are provided to initiate combustion. Unlike the internal combustion engine, combustion is continuous.

The Turbine

As a result of the burning of the air/fuel mixture, the velocity and temperature in the combustion chamber increase rapidly and the gas is forced out of the rear of the engine, through the turbine. The turbine consists of one or more stages of alternate rotating and stationary aerofoil-section blades. It is attached by a shaft to the compressor, and its function is to absorb enough energy from the gas stream to keep the compressor rotating at its optimum speed.
The complete rotating assembly - compressor, shaft and turbine - is carried on bearings and is known as a "spool". In a multi-spool engine, each compressor is driven by one or more turbine stages.

In the turbojet and turbofan, the turbine is designed to absorb just sufficient energy from the gas stream to drive the compressors, leaving the remainder to provide the thrust. The turboprop and turboshift, however, have an additional turbine which is designed to absorb as much energy as possible from the gas stream in order to drive the propeller or power output shaft.

Gas stream temperatures in the combustion chamber can reach 1400 - 1500°C. However, nickel-based superalloy gas turbines run at 1,050 - 1,100°C, approximately the limit for metallic alloys. Sophisticated air cooling techniques are required to maintain this temperature where a fraction of air from the impeller is diverted through to the high pressure turbine disc and blades via a complex network of passages, thereby bypassing the combustion chamber. This results in reduced engine efficiency. An engine that operated at 1400°C would be more efficient, and could use poorer fuels causing less pollution to the environment.

**Sinter Hipped Silicon Nitride**

A sinter hipped silicon nitride high pressure turbine disc, and blades (Figure 2.8a) could provide the solution as the material is stable up to 1,800°C. However in service conditions, the most hostile in the engine (high thermal/mechanical stresses, corrosive environment), (Figure 2.7b,c), even a 1,300 - 1,400°C temperature may still be a limitation for a HIP monolithic. Eventually a reliable material could be found in a HIP sintered silicon nitride-SiC composite (Devendra and Syers 1990). A HIP monolithic material could be used however, for the shroud ring, which encompasses the turbine assembly and is designed to maintain the gas flow over the turbine blades. Superalloys are currently used, but an allowance has to be made for expansion during operation. A smaller gap between the blade tip and the shroud ring leads to greater efficiency and therefore lower specific fuel consumption. HIP silicon nitride provides greater dimensional stability (low thermal expansion coefficient, low creep) and higher temperature capability. A further application
Figure 2.8a HIP Silicon Nitride blades held in a super-alloy disc

Figure 2.8b A range of HIP Silicon Nitride components
could be at the high pressure gas bearing which operates at 400°C -500°C and is situated behind the high pressure turbine. Previous oil lubricated bearings have been limited to temperatures up to 250°C. The bearing consists of a ring which rotates around a rotor journal fixed to the shaft. The ring is manufactured in 4 shells which overlap. This arrangement allows the shells to rock into position during engine start-up thereby creating greater air pressure. At rest the shells lie in perfect cylindrical formation and are initially in contact with the journal. As the speed increases the air pressure builds up, but the shells remain in contact with the journal. Once a speed of around 1500 rpm is reached the shells reach a conformal "Venturi" position and act as a Venturi-like compressor. It is now operating as a gas bearing. The aerodynamic cushion greatly reduces friction and eliminates lubrication problems, and has significantly improved AGT engine performances with a 1-2% reduction in fuel costs in certain cases. Originally the shells were made from Ni-based materials plasma spray coated with chrome oxide. They were then manufactured in silicon nitride around 8 years ago. The journal is made of a Ni-based superalloy and is coated with chrome oxide. New materials need to operate at higher temperatures and have greater resistance to wear caused by friction during start-up and wind down of the engine. Current research continues to be carried out on manufacturing silicon nitride shells to run on a plasma sprayed rotor journal. Other applications for the high temperature, high stress monolithic ceramic are in "cool" areas of the turbine engine, i.e. compressor, and turbocharger rotors, components in piston engines, cutting tools, bend bars and welding nozzles. A few examples are shown in Figure 2.8.

High strength applications also include ball bearings where the light weight and wear resistance properties are suitable for aerospace and electronic applications. High purity, high wear resistant applications include use as a grinding medium for high purity silicon nitride powder grinding.

Industrial applications for reaction bonded, sintered reaction bonded, sintered and hot pressed silicon nitride, and sialon are detailed in Appendix A. They principally encompass metal processing equipment, jigs and fixtures also for induction
heating and vacuum brazing applications, chemical processing equipment, rocket and military weaponry, cutting tools, gas turbine and reciprocating engine components.
PART A
CHAPTER 3. ELEMENTAL ANALYSIS OF PROCESSING FLAWS IN 5 WT% Y₂O₃ SINTER HIPPED SILICON NITRIDE

SUMMARY

Chapter 3 is concerned with the investigation and determination of the elemental composition and origin of processing flaws that were discovered in the sinter hipped silicon nitride ceramic supplied for this research project.

3.1 INTRODUCTION

The introduction describes what form processing defects/flaws generally take, how they are formed during fabrication and how they may affect a material's mechanical and thermal properties.

3.2 EXPERIMENTAL PROCEDURE

This section details how an innovative and effective optical light transparency method was used to research two types of flaw found in the supplied ceramic. The "Cellular Network" and "Metallic Agglomerate" flaws (together with a fabrication encapsulation failure problem) were also examined using reflective optical microscopy and various electron microscopy techniques which are described.

3.3 EXPERIMENTAL RESULTS

The success of the transparency method, and the success of some of the other techniques is discussed in the results. A three dimensional cellular network with an average cell size of 200µm - 2.1mm was identified, as well as clusters of 1 - 3µm metallic particles. These metallic agglomerates ranged from 5 - 45µm in size.

3.4 DISCUSSION

Suitable theories on the reasons for the formation of the flaws are proposed in the discussion, which also includes ideas for further work.
3.5 CONCLUSIONS

While the improvement of material performances by modifying the microstructure progresses the need for manufacturing companies to minimise impurity defects is highlighted.
PART A
CHAPTER 3. ELEMENTAL ANALYSIS OF PROCESSING FLAWS IN 5 WT% Y₂O₃ SINTER HIPPED SILICON NITRIDE

3.1 INTRODUCTION

Processing defects include voids, large grains, inclusions and shrinkage cracks. There are various opportunities for their formation in the fabrication route. In the initial stages the powder purity, size distribution and morphology are contributory factors. For example if a powder needs spray drying, the process may cause the fine particles to form agglomerates. To avoid fabricating an inhomogeneous material a powder of separate fine particles must again be achieved, and this is done by ball milling. Particles are roughly spherical in shape as this allows closer packing, but if some areas are poorly packed then flaws may be created during sintering. Inclusions appear either as unwanted impurities or segregated sintering additives. Their influence in initiating failure varies according to the thermal contraction mismatch or the inclusion toughness relative to the ceramic matrix (Evans 1984). Voids or cavities contain a stress distribution around the perimeter, which can interact with statistically distributed microcracks in the material and cause failure. An example of void formation can be seen in injection moulding processes. A powder/plastic mixture allows injection into a complex-shaped die to produce a component such as a turbine blade. The polymer binder must be removed before sintering and is 'burnt out'. However, this process may leave areas which will subsequently form voids even if the material undergoes a presinter treatment.

Occasionally more severe fabrication defects occur which, due to their size, are immediately noticed on inspection. Figure 3.1 shows the cross-section of a 5 wt% Y₂O₃ SRBSN (HIP) billet with a large area of glass contamination at the centre. During the Green stage of processing, a surface crack was formed which allowed the encapsulating glass to seep in during hipping. The material was originally pressureless sintered before hipping, and this is believed to leave the material susceptible to damage before final densification. Small processing or machining defects can sometimes cause failure during handling or
Figure 3.1 Cross-section of a 5% wt $\text{Y}_2\text{O}_3$ SRBSN (HIP) billet containing glass encapsulant at the centre which seeped in through a crack during hipping
preparation before a component is used for its intended purpose. Figure 3.2 shows the cross-section of a 50mm long transverse ground bar that fractured when lightly clamped for diamond cutting. The series of circular lines in Figure 3.2a are ridges with the highest near the centre. At the machined surface this ridge extends under two small semi-elliptical sub-surface flaws (Figures 3.2b, 3.2c), from which fracture originated (see Section 4.6).

The effects of processing flaws on a material are complex as local stresses due to differences in thermal contraction or in elastic modulus may develop within, or around the flaws. These local stress concentrations are believed to activate microcracks that exist in the vicinity of the flaw and thus produce a crack that subsequently or concurrently initiates the failure. The microcracks are distributed throughout the material and consequently the fracture process exhibits relatively wide statistical variability (Evans 1984, Govila 1988).

3.2 EXPERIMENTAL PROCEDURE
3.2.1 Elemental Segregation in a 5 wt% Y₂O₃ Sinter Hipped Billet (August 1987 HIP Run)
3.2.1.1 Identification of Cellular Network Processing Flaw
A 5 wt% Y₂O₃ sinter hipped silicon nitride billet, fabricated in a HIP run at ASEA in August 1987, was found to contain an elemental inhomogeneity within the bulk volume. A cross-sectional slice of the billet showing a cellular network was prepared by polishing the surface with 6μm diamond compound on a stationary tin lap (see specimen preparation procedures, Appendix A). Using reflection optical microscopy the white speckled phase apparent in cross-sections of the billet was recorded. Normally, transmission optical microscopy is not used to examine silicon nitride ceramics as there is a lack of resolution due to the interference from overlapping grains. However, it was found that at a thickness of 200μm or less, macroscopic details were observed clearly with well defined contrast against the background silicon nitride matrix. A series of experiments were carried out to determine the elemental composition of the cellular network.
Figure 3.2a  Circular shaped ridges on fracture surface.

Figure 3.2b  Top of highest ridge at machined surface

Figure 3.2c  Origin of fracture

Figure 3.2  Cross-section of a transverse machined bar showing fracture surface resultant from a compressive stress
3.2.1.2 **Energy Dispersive X-Ray Analysis (EDX)**

It was thought most probable that the cellular network consisted of differences in the intergranular phase. An area of the cellular network was selected for examination by implementing four small indentations on a carefully polished 3 mm diameter disc, setting out a square area 760µm x 760µm (Figure 3.3). A further two indentations were implemented; Indentation 1 in the centre of an intense area of the cellular network (Area 1) and Indentation 2 in an area free of the structure (Area 2).

Using a 35CF JEOL electron microscope an EDX analysis of various elements was made at the two areas. As iron impurities were found in the form of precipitate agglomerates in the ceramic (Section 3.3.2), this element was considered in the analysis, including Y, N and O levels which constitute the two stable binary eutectic intergranular phases, yttrium disilicate Y₂O₃(SiO₂) and apatite Y₅(SiO₄)₃N.

An area 60 µm x 60 µm was analysed for Fe and Y using a 25kV electron beam with a linear 400s raster scan at a magnification of 1000x and an absorbed current of 0.15 x 10⁻⁹A.

3.2.1.3 **Back Scattered Electron Analysis**

If the cellular network did consist of a different chemical composition then it may be identified by using backscattered electrons which highlight elemental atomic number contrast. A Cambridge S250 electron microscope was used to examine the selected square area (Figure 3.3) in the backscattered mode.

3.2.1.4 **Energy Dispersive X-Ray Dot Map Analysis**

Using a 35CF JEOL electron microscope an EDX two dimensional dot map analysis was made of the selected square area (Figure 3.3). The distribution of Yttria was analysed by setting a window on the Y peak in the spectrum given by a 25kV electron beam, at a magnification of 86 x, and linearly scanning the area in rasters for a 400 s period. The Y Kα peak was not chosen as it resides close to the silicon peak, and consequently overlaps with it. Therefore the Y Kβ peak was used.
Figure 3.3 Transmission optical micrograph of cellular network showing selected area
3.2.1.5 Energy Dispersive X-Ray Digimap Analysis

The two-dimensional relative density distribution of a multi-element composition can be recorded by employing the same EDX technique as in Section 3.2.1.4. However, in this case windows can be placed on a number of elements, and the scan time is increased to 2 hours, which gives a dwell time of 200 ms. This allows a greater number of X-rays to be detected from one spot, therefore giving a stronger signal. The digital data is stored in a computer, and can then be recalled and manipulated to highlight certain features. If the density distribution of one element is required the spectrum for that element is recalled from the computer memory to the V.D.U. (Figure 3.4). If the spectrum has a low count it can be spread by multiplying along the energy axis. Any background counts are eliminated by setting a threshold limit on the lower energy range. When a 'clean' spectrum is achieved it can be offset along the energy axis where equidistant energy bands are assigned to a shade of colour, starting from blue for low energies, to green, yellow, red and white for high energies. Finally the two-dimensional density distribution of the element is set on the V.D.U. and recorded on film. This process is repeated for each individual chosen element. If a multi-element distribution is required then each manipulated spectrum is assigned a different colour and the distributions are set on the V.D.U. An area 200μm x 200μm with its centre 100μm above indentation d (Figure 3.3) was digitally scanned at a magnification of 320 x with a 25kV electron beam. The absorbed current was $1.0 \times 10^{-9}A$ and the dwell time was 200ms.

3.2.1.6 Details of April 1988 HIP Run

Similar to the August 1987 HIP run at ASEA, the April 1988 HIP batch used for machining also contained processing flaws. However, due to the unavoidable delay in material supply and the urgency in directly studying the effects of diamond machining, a very short analysis was made of the existent processing defects using the same examination techniques.

3.2.2 Macro Aggregation of Impurity Phases in a 5 wt% Y$_2$O$_3$
Sinter Hipped Billet (August 1987 HIP Run)

3.2.2.1 Identification of Metallic Agglomerates

The billet containing the cellular network, was also found
Figure 3.4 Energy vs No. counts histogram
to contain metal bearing precipitates within the bulk volume in the form of agglomerates of up to 45\(\mu\)m in size. Using transmission optical microscopy thinned cross-sections of the billet were shown to contain a random distribution of optically dense features.

A series of experiments using analytical electron microscopy were carried out to conclusively determine the elemental composition of the precipitates and the surrounding ceramic matrix.

### 3.2.2.2 Energy Dispersive X-Ray Spot Analysis

Using a JEOL 35 CF electron microscope a number of metal precipitates from different parts of the billet cross-section were analysed with a 15kV electron beam.

### 3.2.2.3 Energy Dispersive X-Ray Digimap Analysis

Using the same EDX digimap technique as described in Section 3.2.1.5 the agglomerate shown in Figure 3.21a was analysed. A total area 50\(\mu\)m x 50\(\mu\)m was digitally scanned with a 15kV electron beam at a magnification of 320 x with an absorbed current of 1.0 \(\times 10^{-9}\) A.

### 3.2.2.4 Details of April 1988 HIP Run

Thinned specimens were viewed with transmission optical microscopy as discussed in Section 3.2.1.1.

### 3.2.3 HIP Glass Encapsulation Failure

The flat as-sintered end of the billet was found to contain a rough texture, and this was examined by optical and SEM microscopy.

### 3.3 EXPERIMENTAL RESULTS

#### 3.3.1 Elemental Segregation in a 5wt\% Y$_2$O$_3$ Sinter Hipped Billet (August 1987 HIP Run)

##### 3.3.1.1 Identification of Cellular Network Processing Flaw

Using reflection optical microscopy the white speckled phase apparent in cross-sections of the billet is seen to increase in intensity the greater the distance from the end of the billet (Figure 3.5a,b and 3.6a). The white speckled phase now appears as a light scattering dark phase using transmission optical
Figure 3.5 Reflection optical photographs of the cross-sections at a) Z = 1.7mm
b) Z = 3.0mm from the as sinter-hipped surface
Figure 3.6a Reflection optical photograph of cross-section at $Z = 8.0\text{mm}$ from the as-hipped surface.

Figure 3.6b Transmission optical photograph of the 21.1mm cross-section $Z = 8.0\text{mm}$ showing light absorbing dark phase.
microscopy (Figure 3.6b). Examination at a higher magnification reveals the dark phase to be a nebulous permeation in the form of a cellular network (Figure 3.7), in which the cell size distribution is large compared to the microstructure. The cellular network structure is three dimensional with an equivalent average cell size of around 400μm in all three planes (Figure 3.8).

3.3.1.1 ii) Surface Pull-Out Enhancement

Using reflection optical microscopy it is shown that the appearance of the cellular network is enhanced due to the contrast caused by a difference in surface topography, which exists in the form of grain pull-out. The grain pull-out is caused by the mechanical tin lap polishing. Figure 3.9a shows the contrast formed by using light incident normal to specimen surface. By tilting the specimen so that the illuminating light was incident at an angle to the surface the image contrast was reversed, producing a bright field image (Figure 3.9b). The higher level of surface pull-out was confirmed with scanning electron microscopy (Figure 3.10).

3.3.1.2 Energy Dispersive X-ray Analysis (EDX)

The spectra obtained from the 60μm x 60μm area (Figure 3.11) show no detectable difference in Y levels between the two areas, but Area 2 shows a trace of Fe. This is probably due to an iron bearing precipitate as this is an area devoid of the cellular structure.

An equivalent area of 60μm x 60μm was analysed to detect any difference in the N and O levels, using a 15kV electron beam. No detectable difference was found between Area 1 and 2. Decreasing the accelerating voltage to 10kV and increasing the absorbed current to 0.3 x 10^-9A again did not reveal any detectable differences (Figure 3.12). A smaller area of 20μm x 20μm was tried but this too did not give a result. If there was a difference in N levels between the two areas it would not be surprising if this were not revealed in the spectra, as the N in the Si grains would dominate and cause a masking effect. This is also true for O and Y, as these are masked by the overall intergranular phase.
Figure 3.7 Transmission optical micrograph of cellular network
Figure 3.8 Three dimensional structure of cellular network. View is a 90° edge, where the billet cross-section was cut along the length of the billet.
Figure 3.9 Reflection optical images of cellular network in dark field and bright field illumination
Figure 3.10 Secondary electron images of cellular network 49° tilt
Figure 3.11 X-ray spectra of Areas 1 and 2
Figure 3.12 Light element X-ray spectra of Areas 1 and 2
Therefore individual pockets of the intergranular phase were analysed at the two areas to avoid this problem. Again, no detectable differences were found (Figure 3.13), and other problems due to beam spreading made the data difficult to interpret. Several β-grains were also analysed but due to the fine grain size, beam spreading did not allow accurate analysis.

3.3.1.3 Back Scattered Electron Analysis

A contrast in the form of dark features is visible in the centre of the selected region (80 x magnification) at Area 1 (Figure 3.14a). At a higher magnification of 160 x this is enhanced and the features can be resolved in the form of curved and straight dark regions (Figure 3.14b). Magnifying these features further at 400 x it is apparent that they consist of small pores which appear as black speckles (Figure 3.14c). The surfaces pores exist in the grey β-grain regions where there is no intergranular phase. However, it is also possible that the pores constituted areas of intergranular phase which had subsequently been pulled out during the surface preparation.

3.3.1.4 Energy Dispersive X-ray Dot Map Analysis

Using the Y Kβ peak for analysis the resultant distribution in Figure 3.15 was obtained. No pattern was observed. Thus another 400s scan was taken on exactly the same area. The dot distribution did not match, proving the scatter at this magnification was too great and a random effect was being recorded. Scans at higher magnification at Areas 1 and 2 were tried, but no differences were observed.

3.3.1.5 Energy Dispersive X-ray Digimap Analysis

The intensity distribution of silicon from the X-ray digimap scan can be seen in Figure 3.16. No particular feature stands out though it is possible a faint circle a, formed by the higher intensity red dots in the top centre may constitute the small circle s in the transmission optical micrograph (Figure 3.3). Comparing reduced Si and Y distributions (Figures 3.17a, 3.17b) a 'hole' A devoid of these elements appears in the same area as in Figure 3.16. The Fe, Cr and Ni distributions did not reveal any patterns.
Figure 3.13 Light element X-ray spot analysis of the intergranular phase
Figure 3.14 Backscattered electron images of the selected area showing topographical contrast
Figure 3.15  Y dot map of selected area
Figure 3.16 Density distribution of Silicon
Figure 3.17a

Figure 3.17b

Figure 3.17  Density distribution of a) Si  b) Y
3.3.1.6 Details of April 1988 HIP Run

Using reflection optical microscopy the white cellular network was recorded and the dimensions measured (Figure 3.18). The network is also three dimensional, but the cell size is larger than for the 1987 HIP run material, ranging from 1.7mm to 2.9mm, with an average size of around 2.1mm.

3.3.2 Macro Aggregation of Impurity Phases in a 5wt % Y₂O₃ Sinter Hipped Billet (August 1987 HIP Run)

3.3.2.1 Identification of Metallic Agglomerates

Using transmission optical microscopy thinned cross-sections of the billet were shown to contain a random distribution of optically dense features with sizes ranging from 5µm-45µm.

A typical dark feature shown in the transmission mode in Figure 3.19a) is revealed to consist of an agglomerate of inclusions in the reflection mode (Figure 3.19b). Due to the optical transparency of the material part of the light is transmitted into the specimen therefore creating a combined reflection and transmission optical image. The dark area represents deeper layers of the cluster under the specimen surface.

3.3.2.2 Energy Dispersive X-ray Spot Analysis

Analysing a number of the metallic precipitates with a 15kV electron beam it was found that each one consistently gave traces of Si, Cr, Fe and Ni; a typical spectrum is shown in Figure 3.20. Fe, Cr and Ni are elements that should not be present in the ceramic and investigations as to the introduction of the impurities led to the source being traced to the ball milling process of the silicon nitride starting powder, where a stainless steel ball mill was used.

3.3.2.3 Energy Dispersive X-ray Digimap Analysis

Figures 3.22a, 3.22b and 3.23a show the density distributions of Fe, Cr and Ni respectively obtained from the 50µm x 50µm area analysed around the agglomerate shown in Figure 3.21. The positions of the precipitates are consistent and the shape of the precipitate P, in Figures 3.21a and 3.21b is exactly reproduced in the Fe and Cr digimaps. In Figure 3.23b the three elements are represented in one multi-element digimap. Precipitate P is again well defined.
Figure 3.19a Transmission optical micrograph of agglomerate

Figure 3.19b Reflection optical micrograph of agglomerate intersecting the specimen surface
Figure 3.21a Reflection optical image of agglomerate

Figure 3.21b Backscattered electron image of agglomerate at a higher magnification
Figure 3.22 Density distribution of a) Fe  b) Cr
Figure 3.23a  Density distribution of Ni

Figure 3.23b  Multi-element digimap showing distribution of Fe, Cr and Ni
A SEM image of the agglomerate and surrounding area appears to show a lack of intergranular phase within the agglomerate region (Figure 3.24a,b). Using Rutherford back scattered electrons and imaging in the topographical mode, the surface texture of the agglomerate area can be examined in detail (Figures 3.25a,b). The precipitates appear as shallow depressions, but over all the surface there are very small "scratch" marks, these being formed by the preferential polishing of the intergranular phase by the soft polishing pad used in the final specimen preparation stage. At the centre of the agglomerate there is just a smooth area with no scratch marks. The appearance of the precipitates as depressions (Figure 3.26) shows that the metallic phase is relatively soft as compared to the silicon nitride phase. The lack of intergranular phase in the agglomerate region is confirmed by the Y digimap (Figure 3.27a) where a 'hole' exists at the agglomerate site. Selecting Fe to represent the agglomerate precipitates, a multi-element digimap showing the distributions of Si, Fe and Y is seen in Figure 3.27b; the hole can now be viewed with respect to the position of the precipitates. No Y is found to be present within the precipitates, and this is confirmed by the X-ray spot analysis results in section 3.3.2.2. This suggests that the lack of Y in the agglomerate region is not due to a precipitate absorption but an exclusion of the intergranular phase from the region. A further analysis comparing O and Fe sites also showed no overlap.

3.3.2.4 Details of April 1988 HIP Run

Metallic agglomerates similar to the ones found in the August 1987 HIP run are smaller in size, ranging from 3μm - 14μm, with a few reaching 25μm (Figure 3.28). Their presence is unexpected, as the previous ones constituted steel particles which had been introduced during the silicon nitride powder ball milling stage. The present batch was supposed to have been milled with a silicon nitride ball mill. The elemental composition was not analysed and confirmed though.

The fracture origin in a flexural rupture test was found to be due to a processing flaw containing Al and Cl (See Section 6.6). It is probable that while Turner and Newall Ltd changed to a silicon nitride ceramic ball, rather than using a steel one, the type used was probably 102A2 SRB silicon nitride which contains
Figure 3.24a  SEM micrograph of agglomerate and surrounding area

Figure 3.24b  SEM micrograph of metal bearing phase appearing as white areas
Figure 3.25 Rutherford back scattered electron micrographs taken in the topographical mode showing agglomerate precipitates and the preferential polishing of the intergranular phase
Figure 3.26 Shallow angle view of agglomerate. Precipitate P is clearly defined in the centre. The slight depression is due to preferential polishing of the softer metallic phase.
Figure 3.27a Density distribution of Y

Figure 3.27b Multi-element digimap showing distribution of Fe, Si and Y
Figure 3.28a Transmission optical micrograph of a thinned specimen from the April 1988 HIP run batch. The sharp black spots are light absorbing metallic agglomerates.

Figure 3.28b Reflection optical micrograph of same area as in Figure 3.28a showing agglomerate of metallic particles.
10 wt% \( \text{Y}_2\text{O}_3 \), 2 wt% Al and 2wt% Cr sintering additives, and is fabricated by Turner and Newall.

### 3.3.3 HIP Glass Encapsulation Failure

The flat as-sinter hipped end of the billet was found to contain a rough surface texture (Figure 3.29a). Close examination revealed a surface porosity which took the form of large irregular shaped channels and pores (Figures 3.29b, 3.30a,b), and concentrations of regular shaped pores (Figure 3.31). The dimensions of the irregular channels and pores ranged from \((4\text{mm} \times 300\mu\text{m} \text{ to } 1015\mu\text{m} \times 500\mu\text{m})\) and \((1212\mu\text{m} \times 999\mu\text{m} \text{ to } 150\mu\text{m} \times 120\mu\text{m})\) respectively. The size of the regular shaped pores varied from 18\(\mu\text{m}\) to around 500\(\mu\text{m}\), while some areas of the surface contained concentrations of pores around 70 - 80\(\mu\text{m}\) in size. The average depth of the irregular shaped porosity = 13.4\(\mu\text{m}\) \((\pm \sigma = 4\mu\text{m})\), which compares with the average depth of the regular shaped pores = 13.3\(\mu\text{m}\) \((\pm \sigma = 4.8\mu\text{m})\). This does not take into account the surface curvature or "lip" which extends up to 7\(\mu\text{m}\) in height and exists at the rim of most of the porosity. The equivalence in depth shows that the two types of porous deformation derive from the same process.

The extensive porosity on the as-hipped surface is almost certainly due to a failure in the ASEA glass encapsulation technique. The boron nitride parting layer appears to have formed cracks, possibly during drying, therefore allowing the Pyrex ampoule, or powder, to react directly with the billet surface during the sinter hipping process. No information was forthcoming from ASEA.

Many surface channels and pores were found to contain a number of small internal circular pores (Figure 3.32a,b). The diameter of these ranged from 8\(\mu\text{m}\) to 27\(\mu\text{m}\), with an average of 16.1\(\mu\text{m}\) \((\pm \sigma = 4.6\mu\text{m})\), and the depth ranged from 2.0\(\mu\text{m}\) to 8.6\(\mu\text{m}\) with an average of 4.9\(\mu\text{m}\) \((\pm \sigma = 1.7\mu\text{m})\). It is believed that they were formed by escaping nitrogen gas during the hipping process.

Encapsulation techniques used to fabricate highly densified ceramic components should allow a forming process that produces a smooth surface which requires a minimum amount of final machining. The surface roughness in non-porous areas was
Figure 3.29a  Optical photograph of the as-hipped surface showing surface porosity. 0° tilt

Figure 3.29b  SEM micrograph of an irregular-shaped channel. 46° tilt
Figure 3.30a

Figure 3.30b

Figure 3.30  SEM micrographs of irregular-shaped voids. 45° tilt
Figure 3.31 SEM micrographs of regular-shaped voids. 45° tilt
Figure 3.32a  SEM micrograph of an irregular-shaped void containing a small internal pore. 45° tilt

Figure 3.32b  SEM micrograph of a small symmetrical void. 45° tilt
measured with a Talystep stylus instrument (See Appendix B.3) and the centre line average roughness was estimated to be 1.0μm. In certain small areas 100μm x 100μm this figure dropped to 0.3μm. This is probably the surface texture produced with a good encapsulation technique.

On all the as-hipped surface there exists a needle-like structure which is unidirectional in small areas, but each area contains a different orientation. The void in Figure 3.33a exists in an area where the orientation travels from bottom right to top left. Magnification of the area reveals a hollow feature (Figure 3.33b,3.34) which is also present on all areas of the surface, but generally smaller in size. The needle-like structure and the hollow impression may be formed by the sand blasting removal process of the encapsulating glass. Sand blasting is a surface "stripping" technique where hard particles are driven on a surface at high velocities. The hollow impression may have been caused by a larger than average size particle. Great care should be taken with this removal process as surface damage similar to machining damage can be formed. However another cause may have been the structure of the material that was finally in contact with the silicon nitride during hipping.

3.4 DISCUSSION
3.4.1 Elemental Segregation in a 5 wt% Y2O3 Sinter Hipped Billet
3.4.1.1 Techniques Used
EDX analysis on large areas will not have the sensitivity to resolve any differences between a cellular network region and clear region due to the masking effect of the general matrix. It is almost certain that the composition of the network exists in the crystalline intergranular phase and not in the SiN grains as substitution of atoms in the covalently-bonded Si3N4 tetrahedral superlattice structure is limited, and only occurs in the α-SiN structure as the hexagonal unit cell has twice the volume with two interstitial sites.

Carrying out EDX spot analysis on pockets of intergranular phase will not resolve the true elemental composition either, due to the problem of beam spreading.
Figure 3.33a  SEM micrograph of needle-like structure.  
45° tilt

Figure 3.33b  Hollow feature situated inside the void.  
45° tilt
Figure 3.34 High magnification micrograph of hollow feature. 45° tilt
Backscattered electron analysis may not have resolved atomic number differences but it has been successful in revealing the surface porosity which appears to exist in areas of the cellular network (Figures 3.9 and 3.10). This effect has already been noted in a University of Warwick report (Plucknett 1987) where the surface of a high glass fraction 10% wt Y$_2$O$_3$ sinter-hipped silicon nitride specimen suffered grain pull-out in certain isolated areas approximately 0.5-1mm in size when polished. Plucknett also found that the intergranular phase (glass volume) was not identical throughout the bulk ceramic and rose to 18-19 vol% in isolated areas, possibly constituting the areas of weakness.

A similar observation was made in work carried out at Rolls-Royce Bristol by Dr A Dunhill, where features on a HPSN ball bearing were highlighted by using ultrasound. Circular rings of cell size approximately 1-2mm were found on the surface. It is thought that they were pores formed in high glass fraction areas and enhanced as a result of machining. Their formation may have been due to agglomerates of particles 1-2mm in size that sintered and densified to a higher than normal density, thus creating a balancing less densified higher glass fraction outside.

It is not surprising that a dot map analysis did not resolve the composition of the network as its structure is diffuse on a macro scale. Even using an EDX digimap analysis where the electron beam advances every 200ms a definite elemental difference is not conclusively detected. An interesting note however, is that small circles with slightly higher concentrations of Si and Y (Figures 3.17a,b) may constitute the higher intergranular phase volume areas.

3.4.1.2 Origin and Response to Applied Loading

The formation of the cellular network was at first attributed to the phenomenon of constitutional super-cooling as a few of the characteristics proved to be consistent with the theory. However rather than columnar cells being present the network was found to consist of a three dimensional structure, with the same cell size in three planes.

The formation of the flaws could be caused by the roughly
spherical powder particles closely compacting together in some areas to form highly dense regions, while the poorly packed areas subsequently form flaws. However, this would create a random flaw distribution. It is also believed that variations in the crystallinity of the glassy phase in ceramics nucleates at certain sites which then spread, either leaving behind or pushing out a slightly modified intergranular phase, which will be imperceptible to detect on a micro-scale.

Tests on HPSN containing light-coloured circular rings by Dr A Dunhill of Rolls-Royce Bristol, led to an observation that agglomerates of particles 1-2mm in size closely compacted together during sintering leaving circular less densified higher glass fractions outside. This appears to be consistent with the observations in 5 wt% Y_2O_3 sintered material, although the intergranular phase is crystalline. Similar effects have been seen in other types of sintered silicon nitride (Syalon 201, GTE AY6, T and N LTD 10.2A.2), although they are described in reverse. The dark grey or black spots existing within the lighter grey bulk of the material are commonly called the "black death" of "the pox". Therefore, it is clear that the formation of the cellular flaws is independent of the type of sintering additives, or the structure of the subsequent intergranular phase, and therefore must depend on local interactive effects between the particles in the powder.

Kendall et al (1989) have found that a phenomenon called flocculation clustering occurs in materials formed in colloidal systems. In an experiment using laser light scattering for particle size measurement, dominant particle clusters of 60µm size were consistently formed in T_iO_2 during drying, pressing chemical destabilizing, centrifuging or redispersing of a fine colloidal suspension containing no particles >2µm in diameter. The theoretical analysis of the stable clusters is based on the competing processes of growth and breakdown of the cluster, which is modelled as a close-packed aggregate of equal spheres. Growth is dominated by the external surface energy U_s of the cluster, as in nucleation theory (Binder and Stauffer 1976, Zettlemoyer 1977, Hoare et al 1980, Larson and Garside 1986).

\[ U_s = 5.7 \Delta^2 \gamma_{SL} \quad \ldots \ldots \quad (2.1) \]
where \( A \) = spherical cluster diameter containing \( 0.7 \left( \frac{A}{D} \right)^3 \) particles of diameter \( D \), and \( \gamma_{SL} \) = interfacial tension between the solid particles and the surrounding liquid. Breakdown is determined by the internal bonding energy \( U_i \) resulting from Van der Waals elastic contacts between the particles (Johnson et al. 1971, Kendall and Padget 1982, Kendall et al. 1986). Each particle contact has an energy \( -1.47 \gamma_{SL} \left[ 9 \pi \gamma_{SL} D^2 \left( 1-v^2 \right) / E \right]^{2/3} \), where \( E \) is Young’s modulus and \( v \) is Poisson’s ratio.

Thus the total energy of the cluster is:

\[
U_i = -6.53 \left( \frac{A}{D} \right)^3 \gamma_{SL} \left[ 9 \pi \gamma_{SL} D^2 \left( 1-v^2 \right) / E \right]^{2/3}
\]

\[
\ldots \ldots 2.2
\]

For the dominant cluster size, \( A_0 \) the growth and breakdown energies are balanced, \( d \left( U_i + U_g \right) / dA = 0 \) giving:

\[
A_0 = 0.064 \left[ ED^{2.5} / \gamma_{SL} \left( 1-v^2 \right) \right]^{2/3}
\]

\[
\ldots \ldots 2.3
\]

Equation 2.3 has been validated by Kendall et al. (1989) by particle-size and flaw-size measurements on sintered ceramic specimens. Dominant cluster sizes were predicted and then compared with flaw sizes corresponding to measured low bending strengths. Higher strengths have been achieved in \( TiO_2 \) using different methods to break down the clusters (Alford 1987) or prevent the clusters from forming (Eur. Pat. Publ 1988).

Using this analysis based on weak colloidal forces the dominant cluster size can be estimated for the 5 wt% \( Y_2O_3 \) sintered material where,

\[
E = 322 \text{ GPa} \quad \gamma_{SL} = 0.3 \text{ Jm}^{-2} \quad \text{and} \quad v = 0.33
\]

The minimum and maximum limits of the powder size \( D \) ranged from 0.1 \( \mu \text{m} \) - 4 \( \mu \text{m} \). This would give a dominant cluster size \( A_0 \) of 16 \( \mu \text{m} \) - 10.6 \( \mu \text{m} \), which is comparable within an order of magnitude...
to the 400μm cell size of the August 1987 material and 2.1mm cell size of the April 1988 material (Section 3.3.1.1i and 3.3.1.6). Using the estimated modal value of particle size 0.4μm - 0.6μm the predicted values of 200 μm to 310 μm are even closer to the measured values. Due to a lack of available material no evidence of their influence on the strength of the material was tested for. However, it can be said that once dominating flaws are eliminated from the material they must have some small and limited effect, as enhanced pull-out is caused in the cellular network regions.

3.4.1.3 Further Work

Two further tests could be carried out to determine the composition of the cellular network. The intergranular phase volume can be found in areas containing the network and compared with the clear areas. This apparently has been done by the University of Warwick, although the result was inconclusive. A better technique would be to prepare a TEM specimen and analyse the cellular network area with X-ray spot analysis. With this method the electron interaction in the material sub-surface causing the volume effect or beam spreading, would be eliminated. Therefore an accurate statistical analysis could be made of the elemental composition of pockets of intergranular phase in the cellular network and clear areas. However, practically this would be very difficult as the probability of producing an electron transparent area exactly on an area containing the cellular network would be very low. Even if this were achieved it would be very difficult to know if the electron transparent area actually did contain the network, due to the near invisibility at thickness less that 50 μm and the diffuse appearance when viewed at high magnification in the transmission optical mode.

3.4.2 Macro Aggregation of Impurity Phases in a 5wt% Y₂O₃ Sinter Hipped Billet

The agglomerate region proved to contain no intergranular phase. This suggests that during the sinter hipping process the steel particles melted and partially dissolved in the matrix. The high concentration of Fe rich inclusions reduced the local medium viscosity and therefore on cooling, local concentrations of the metal rich solution in the matrix precipitated out into
an agglomerate of precipitates, while excluding the formation of the intergranular phase. After discussions between Rolls-Royce and the ceramic supplier, Turner and Newall Limited, it was decided that a steel ball mill would no longer be used, and would be replaced by one made of silicon nitride.

It is difficult to ascertain the effect of the different expansion coefficient of the inclusions relative to the matrix. It appears that the inclusions are compliant and remain attached to the matrix (Figure 3.26), and this can give rise to several modes of failure (Evans 1984).

Due to a lack of available material, 3 point flexural rupture tests could not be carried out by Mr R Quinn at Rolls-Royce Leavesden. However, other tests carried out on sintered reaction bonded silicon nitride containing 10 wt% Y2O3, 2 wt% Al and 2 wt% Cr sintering additives (102A2 SRBSN), highlighted the problem of processing flaws (Quinn 1989). Fractographic analysis of flexural rupture test bars machined to different surfaces finishes showed that failure was mainly due to processing flaws which took the form of iron and chromium silicide agglomerates up to 100 µm in size, voids of 100 - 200µm diameter, and regions of incomplete densification. Their effect was most critical on fine machined samples of 0.05µm/0.1µm CLA roughness, where only 15% of failures occurred from surface originating machining defects. Similar weakening effects have also been found by Govila in fracture tests on SSN with oxide additives (Govila 1985 and 1988 and Larsen and Adams (1984) on a range of other commercial materials (Table 3.1).
<table>
<thead>
<tr>
<th>Material</th>
<th>Density (g/cm$^3$)</th>
<th>Bend Strength (MPa)</th>
<th>Grain Size Average (μm)</th>
<th>Maximum (μm)</th>
<th>Fracture Origins</th>
</tr>
</thead>
<tbody>
<tr>
<td>Norton NC-132 (13%MgO)</td>
<td>3.18</td>
<td>710</td>
<td>0.5-1.0</td>
<td>3.0</td>
<td>Primarily machining flaws, other processing defects</td>
</tr>
<tr>
<td>Norton NCX-34 (82%Y$_2$O$_3$)</td>
<td>3.37</td>
<td>673</td>
<td>0.5-1.8</td>
<td>6.0</td>
<td>Machining flaws and processing defects</td>
</tr>
<tr>
<td>Harbison-Walker (TO Ceria)</td>
<td>3.33</td>
<td>529</td>
<td>0.5-2.5</td>
<td>Primarily inclusions</td>
<td></td>
</tr>
<tr>
<td>Kyocera SN-3 (42%MgO, 5%Al$_2$O$_3$)</td>
<td>3.07</td>
<td>516</td>
<td>1.0-3.0</td>
<td>12-17</td>
<td>Primarily inclusions</td>
</tr>
<tr>
<td>CERADYNE Ceralloy 147A (12%P)</td>
<td>3.22</td>
<td>600</td>
<td>0.5-3.0</td>
<td>14</td>
<td>Undetermined</td>
</tr>
<tr>
<td>CERADYNE Ceralloy 147Y (13%Y$_2$O$_3$)</td>
<td>3.37</td>
<td>605</td>
<td>1.0-3.0</td>
<td>10-12</td>
<td>Machining flaws and processing defects</td>
</tr>
<tr>
<td>CERADYNE Ceralloy 147Y-1 (18%MgO)</td>
<td>3.23</td>
<td>573</td>
<td>0.5-1.8</td>
<td>5-7</td>
<td>Dark inclusions</td>
</tr>
<tr>
<td>Fiber Materials, Inc. (46%MgO)</td>
<td>3.17</td>
<td>450</td>
<td>0.5-1.8</td>
<td>5-7</td>
<td>Dark inclusions</td>
</tr>
<tr>
<td>Toshiba (42%Y$_2$O$_3$, 3%Al$_2$O$_3$)</td>
<td>3.25</td>
<td>729</td>
<td>0.5-2.0</td>
<td>Dark, shiny inclusions</td>
<td></td>
</tr>
<tr>
<td>Toshiba (30%Y$_2$O$_3$, 30%Al$_2$O$_3$, 30%SiO$_2$)</td>
<td>3.20</td>
<td>576</td>
<td></td>
<td>Inclusions</td>
<td></td>
</tr>
<tr>
<td>Westinghouse (42%Y$_2$O$_3$, 50%P$_2$O$_5$)</td>
<td>3.26</td>
<td>627</td>
<td>0.5-2.0</td>
<td>Dark, shiny inclusions</td>
<td></td>
</tr>
<tr>
<td>NASA/AVCO/Norton (15%MgO)</td>
<td>3.37</td>
<td>629</td>
<td></td>
<td>Dark, shiny inclusions, other processing defects</td>
<td></td>
</tr>
<tr>
<td>Battelle HIP (55%Y$_2$O$_3$)</td>
<td>3.25</td>
<td>620</td>
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</tbody>
</table>

### Sintered Si$_3$N$_4$

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (g/cm$^3$)</th>
<th>Bend Strength (MPa)</th>
<th>Grain Size Average (μm)</th>
<th>Maximum (μm)</th>
<th>Fracture Origins</th>
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</thead>
<tbody>
<tr>
<td>Kyocera SN-20S (53%MgO, 9%Al$_2$O$_3$)</td>
<td>2.91</td>
<td>260</td>
<td>0.5-1.0</td>
<td>4.0</td>
<td>Inclusions and porosity</td>
</tr>
<tr>
<td>Kyocera SN-201 (45%MgO, 7%Al$_2$O$_3$)</td>
<td>3.00</td>
<td>342</td>
<td>0.5-2.0</td>
<td>4.0</td>
<td>Primarily inclusions, some pores</td>
</tr>
<tr>
<td>GTE Sylvania (60%Y$_2$O$_3$)</td>
<td>3.23</td>
<td>537</td>
<td>0.5-3.0</td>
<td>5-8</td>
<td>Inclusions, pores and pore/inclusions</td>
</tr>
<tr>
<td>ATR Research (23%Y$_2$O$_3$, 40%A1$_2$O$_3$)</td>
<td>3.10</td>
<td>547</td>
<td>0.5-2.0</td>
<td>6.0</td>
<td>Surface and subsurface porosity</td>
</tr>
<tr>
<td>Rockendyne SN-56 (50%Y$_2$O$_3$, 24%A1$_2$O$_3$)</td>
<td>3.25</td>
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<td>0.5-2.0</td>
<td>8-9</td>
<td>Porosity open to tensile surface</td>
</tr>
<tr>
<td>Rockendyne SN-104, SN-48 (14%Y$_2$O$_3$, 7%SiO$_2$)</td>
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<td>356</td>
<td>0.5-1.5</td>
<td>5-6</td>
<td>Porosity open to tensile surface</td>
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</table>
3.5 **CONCLUSIONS**

Continuous research is carried out by ceramic manufacturers to manipulate the microstructure in order to produce ceramic materials with improved mechanical properties. However, before these advancements have any effect it is vital that processing flaws are eliminated, as in most cases they are a probable source of material failure (Govila 1985 and 1987, Quinn 1989, Table 3.1). The fabrication of an engineering component is a long process, beginning with the production and processing of the constituent powders, followed by their compaction and densification. Various companies are involved at different stages of component production and each one must ensure that the upmost care is taken with cleanliness and material handling. It can be envisaged that a clean room approach will be adopted by all companies associated with ceramic production in the future.
Chapter 4 provides the basis for the reader to understand the theories behind the description of machining damage in ceramic surfaces.

4.1 INTRODUCTION

The introduction highlights the importance of being able to minimise the formation of machining flaws in order to improve a material's performance. It also summarises the work and techniques used by various authors in the description of machining damage, residual stresses and cracks.

4.2 GRIFFITH THEORY OF FRACTURE

The Griffith theory of fracture is initially described as it is the first and still relevant basic theory in the description of crack initiation and propagation in brittle materials.

4.3 SHARP INDENTOR STRESS FIELDS

The theory then moves on to the analysis of sharp indentor stress fields as this helps to understand the complex stress fields, crack initiation and elastic/plastic deformation in a machined surface; indentation crack initiation near field stresses, and propagation far field stresses are discussed.

4.4 INDENTATION FRACTURE – FRACTURE TOUGHNESS AND STRESS INTENSITY FACTOR

Once stress field issues have been covered the concepts of fracture toughness and stress intensity factor are explained as they are used to describe fracture mechanics in brittle bodies.
4.5 INDENTATION CRACK RESPONSE TO APPLIED LOADING

Arriving closer to the analysis of machined surfaces the response of indentation cracks to applied loading is described.

4.6 MACHINING-INDUCED DEFORMATION/FRACTURE AND CRACK RESPONSE TO APPLIED LOADING

Armed with an understanding of the previous concepts, machining-induced mechanical damage is discussed, and in essence it is explained that:
1. The deformation and fracture generated by individual particle contacts during diamond machining resembles the damage associated with sharp diamond indentations.
2. The overlap and interaction of residual stresses from neighbouring damage sites gives rise to a thin surface layer under residual compression, governed by a long range biaxial field, which has an underlying and compensating field of residual tension. The effect of these fields and local stress fields on underlying strength-controlling median cracks is also discussed. A modified version of Griffith's strength equation for ceramics is derived.

4.7 THE RESIDUAL COMPRESSIVE LAYER - DAMAGE RESISTANCE AND DELAMINATION

The competing process between compressive and tensile fields and the occurrence of surface delamination is discussed.

4.8 STATISTICS OF FRACTURE STRENGTH

The basic theory of statistics of fracture strength is presented, and this allows estimations of fracture strengths derived from flaw sizes to be made later on in the thesis.

4.9 SINGLE POINT SCRATCH TESTS

Finally, the work of various authors on single point scratch tests is summarised and includes details on mechanical deformation/fracture.
PART B  DIAMOND MACHINING IN 5 WT% SINTER HIPPED SILICON NITRIDE

CHAPTER 4. THEORY

4.1  INTRODUCTION

State of the art structural ceramic components are having to meet ever more stringent tolerances in an increasing number of engineering applications. This requires the use of a precision machining process where the resultant mechanical damage is kept to a minimum. This is important as the strength of a component is highly dependant on the integrity of the surface and subsurface layers which will contain a distribution of machining flaws. An understanding of the formation of these flaws may contribute to the refinement of machining procedures, and therefore an improvement towards achieving the said properties of the material.

Of the various machining techniques available for ceramics, the most commonly used for research is the peripheral grinding wheel. The damage introduced by unidirectional hard sharp particles leaves residual stresses that are both compressive and tensile in nature. Two populations of strength controlling cracks are also observed (Mecholsky et al 1977, Rice and Mecholsky 1979), one set normal to the direction of motion of the grinding particles, and the other more severe, set parallel.

Various studies have shown that an under-lying residual tension exists at the site of the strength-controlling damage (Marshall et al 1983, Kirchner and Isaacson 1982 and 1983, Marshall 1984), and this has an important bearing on the strength properties of a material. The influence of the tension has been detected by using acoustic wave scattering to monitor in situ the response of machining - induced cracks during failure testing (Marshall et al 1983), and by measuring strength/crack-size relations in several brittle materials (Marshall and Lawn 1980, Kirchner and Isaacson 1982 and 1983).

The existence of shallow layers in residual compression in machined ceramic surfaces has been observed by destructive and non destructive methods: indentation-fracture in a glass ceramic (Cook 1981) and silicon nitride (Marshall et al 1983), the measurement of the bending of thin plates of silicon nitride machined on one surface (Marshall 1984, Johnson-Walls et al 1986)

Due to the nature of mechanical abrasion, surface damage is not limited only to the grinding regime. Even the action of finely polishing the surface of Al₂O₃ with 0.25 μm diamond particles, residual compressive stresses are formed that have caused severe buckling of thinned TEM foils (Hockey 1972).

Marshall et al (1983) have suggested that the origin of the residual stresses can be understood by regarding machining damage as an accumulation of a large number of isolated sharp particle contact events. An isolated elastic/plastic contact gives rise to a radially compressive residual field, with a tangential tension outside the plastic zone (Marshall and Lawn 1979, Lawn et al 1980). Strength-controlling cracks form on median planes within the tensile field. The overlap of residual fields from neighbouring damage sites in a machined surface gives rise to a shallow layer of compressive stress. This compression tends to reduce, but does not eliminate the residual tension on the strength-controlling flaw (Cook et al 1981, Kirchner and Isaacson 1982 and 1983, Marshall et al 1983, Marshall 1984).

The nature of machining damage is complex and the removal of material can leave a surface with chipping, grain pull-out, cracks and plastic deformation. The strength of a machined ceramic material has been shown to depend on the quality of surface finish (Sedlacek 1972, Wu and McKinney 1979, Andersson and Bratton 1979, Johnson-Walls et al 1986, Samuel and Chandrasekar 1989, Quinn 1989) and this is ultimately determined by the grinding parameters. Therefore considerations must be given not only to the depth of cut, but also to the abrasive type, size, distribution and bond, wheel speed, lubricant, material feed rate and machine stiffness.

The objective of the work was to study the macroscopic and microscopic effects of machining damage caused by diamond grinding in different surface finishes. Using a cross-sectional
technique the machined surface and sub-surface layers were investigated normal and parallel to the machining direction to determine the nature of the compressive layer and the extent of sub-surface crack formation. A comparison would then follow with experimental measurements from flexural strength tests carried out by R Quinn (1989) and X-ray diffraction stress tests by P E J Flewitt (1989).

4.2 GRIFFITH THEORY OF FRACTURE

Early work by Griffith (1920) provided a basis of describing the processes in crack initiation and propagation and their relation to the strength of a material. However, an assumption that materials were defect free led to theoretical strengths calculated on atomic bond strengths being 10 x too high for soft metals measurements and 1000 x too high for brittle material measurements. An explanation was found in strength tests carried out on glass fibres of varying thickness, where a decrease in fibre thickness was accompanied by an increase in strength. This strength dependence on size was attributed to the presence of statistically distributed micro cracks, or Griffith flaws, of which the largest or dominant flaw would develop into a propagating crack under an applied stress. Thus the extension of a well defined crack was described by a reversible thermodynamic process where the total energy of the crack system $U$ is given by:

$$U = (-W_L + U_e) + U_S$$ \hspace{1cm} (4.1)

where $-W_L$ = work done by applied force

$U_e$ = elastic strain energy of the crack

$U_S$ = total surface energy of the crack.

The bracket term $(-W_L + U_e)$ is known as the mechanical energy of the crack system, where dynamic effects are not included. By balancing the mechanical and surface energy terms thermodynamic equilibrium of the crack system is reached. At the crack tip, cohesive molecular forces must be overcome in the formation of new fracture surfaces and therefore the surface energy term must
increase. Strain energy release is largely dissipated by plastic flow.

By including the energy expended in plastic flow Irwin (1958) suggested that Griffith's theory would be generally applicable. Sneddon (1969) showed that the equilibrium requirement in the case of a penny-shaped crack was:

\[
\frac{d}{dA} [-W_L + U_s] + \frac{d}{dA} US = -G + 2\tau = 0
\]

\[\ldots\ldots\ldots\text{(4.2)}\]

taking the z axis as the axis of symmetry and c the crack radius, where \(G\) is the strain energy release rate, \(\tau\) is the fracture surface energy and \(A = \pi c^2\).

The stress intensity factor \(K_I\) represents the stress amplitude at a highly stressed region of a crack in an elastic solid. It is dependent on the size and shape of the crack, the loading configuration and certain geometrical limits. Following the approach taken by Irwin (1958) the strain energy release rate is seen as the work done by applying a tensile stress \(\sigma_{zz}(x, o)\) on the crack faces \(c \leq x \leq c + 5c\) to restore the crack to its original size.

The strain energy is given by:

\[
\delta U_E = \int_{c+5c}^{c} x\sigma_{zz}(x, o) dx
\]

\[\ldots\ldots\ldots\text{(4.3)}\]

Sneddon approximated the stress and displacement with

\[
\sigma_{zz}(x, o) = \frac{K_I}{[2(x-c)]^{1/2}}
\]

\[\ldots\ldots\ldots\text{(4.4)}\]
and

\[ U_z(x, o) = 4 \frac{(1-v^2)}{E} K_I (c+\delta c-x)^{1/2} \] .... (4.5)

giving

\[ G = \frac{1}{2\pi c} \frac{dU_z}{dc} = \frac{(1-v^2)}{2^{1/4} E} K_I \] .... (4.6)

where \( dA \) is set = \( 2\pi c dc \) and \( \delta c \) tends to zero, \( K_I \) is the stress intensity factor. Plane strain derivations in work by Lawn and Wilshaw (1975) differ by the factor \( 1/\sqrt{2} \) and this is due to the choice of reference geometry. Work carried out by Sneddon (1969), Sih (1973) and Lawn et al (1980), led to the formulation of the stress intensity for the half-penny crack configuration:

\[ K_I = f(\Phi) \frac{2}{(\pi c)^{1/4}} \int_c^c \frac{x\sigma(x)}{(c^2-x^2)^{1/4}} dx \] .... (4.7)

where \( f(\Phi) \) is a function that takes account of the free surface and \( \sigma(x) \) is the applied tensile stress.

4.3 SHARP INDENTOR STRESS FIELDS

The understanding of the complex stress fields, crack initiation and elastic/plastic deformation in a machined surface can be aided by considering first of all the stress fields and initiation of fracture associated with a single sharp indentor. (Note: A sharp indentor is defined as one in which the contact is essentially plastic in nature up to the point of fracture). However, even this has proved very difficult to do judging by the number of papers written on this subject and the conflicting theories proposed. A brief summary on the sequential development
of various models will be given, culminating with the approach as proposed by Chiang, Marshall and Evans (1982).

4.3.1 Sharp Indentors

Before the complex problem of stress fields and crack initiation is discussed a simplistic, descriptive account of sharp indentor crack formation will be given, without taking crack initiation into consideration.

The indentor makes contact with the material surface and penetrates, forming a deformed zone around the indentation site. This gives rise to surface tensile zones adjacent to the contact, as well as a sub-surface tensile stress field immediately beneath the indentor tip (Figure 4.1a). A totally contained, sub-surface vent (or penny-like crack) may also be formed immediately beneath the tip of the indentor where sub-surface tensile stresses are greatest (Figure 4.1b). Further loading causes the median vent, or crack, to grow downwards and outwards (Figure 4.1c), although the outward growth is partially restricted on inhibited by near-surface compressive stresses (see also Figure 4.5). Two situations may now occur. For heavily loaded specimens further loading causes the sub-surface median (penny-like) cracks to break through the confining stress lobes and intersect the specimen surface forming half-penny median/radial cracks (Figure 4.1d). Otherwise, for a specimen not as heavily loaded, the median (penny-like) cracks remain sub-surface during the loading cycle, extending downwards and slightly outwards along principal normal stress contours (see Section 4.3.3 Crack Propagation) breaking through to the surface during the unloading cycle when the intensity of the confining compressive stresses has been sufficiently reduced.

Upon unloading the median vents attempt to close up, but are prevented from doing so by debris and the existence of a residual stress field caused by the indented material attempting to accommodate the plastic deformation zone (Figure 4.1e). This residual stress field or elastic/plastic mismatch gives rise to saucer-shaped lateral cracks which originate at the base of the plastic zone and extend in a plane parallel to the specimen surface as the indentor is further unloaded (Figure 4.1f). The
Figure 4.1 Median, radial and lateral crack systems during sharp indentor loading (+) and unloading (−). The black region at each stage signifies a plastic zone. Point loading is shown in Figures 4a and 4g respectively.

The black and unshaded regions in these figures represent tensile and compressive stress fields.
(After Lawn and Marshall (1978))
lateral cracks continue to extend after indentor removal under the influence of the residual stresses (Figure 4.1g) which may also drive partially developed sub-surface median cracks to completed half-penny median/radial cracks. In heavily loaded bodies the lateral vents may intersect the surface causing chipping.

4.3.2 Indentation Crack Initiation—Near Field Stresses

The total stress field present around an indentation is made up of two stresses, "near-field" and "far-field". It is the near-field stresses which determine the initiation point of cracks created by indentation, and these can be described by three components. Radial stresses radiate from the central point of contact, hoop stresses are present around the contact site and shear stresses are the more complex directional stresses. Far-field stresses are also described by the same three components but exist at a distance from the contact region. These determine the propagation of the indentation induced cracks. A maximum tensile stress is formed by the interaction of the three components in both the near and far fields and this effectively controls the fracture initiation and propagation conditions and processes.

Work by Lawn and Swain (1975), where the maximum tensile stress in a sharp contact event was found to occur directly below the indentor point at an elastic-plastic interface, was used in the later model as proposed by Lawn and Evans (1977).

In this model the initiation of median cracks below a sharp Vickers pyramidal indentor in a brittle body is described by considering the deformed region as a plastic zone formed by the radial movement of material displaced and densified by the indentor. A resultant elastic-plastic stress field is analogous to the field as proposed by Hill's expanding cavity model which describes an expanding spherical void subjected to an internal hydrostatic force (Hill 1950, Marsh 1964, Johnson 1970, Lawn and Evans 1977, Puttick et al 1977). This led to the reasoning that the depth and width of the elastic-plastic boundary would increase with higher loads due to the greater plastic zone volume, while the tensile stress at the boundary would remain constant. However, this early model contained an inherent
assumption that there existed fortuitous flaws beneath the contact site that would be searched by the maximum tensile field and then made unstable, forming the sub-surface median vent which would finally grow to the surface median radial crack formation. Later work by Hagan and Van der Zwaag (1984) in fact showed that the initiation of median and lateral cracks in various glass materials could also result from the deformation caused by the indentation process, this also occurring beneath the contact site.

Another potential discrepancy lay in the use of the expanding cavity model which requires a uniform contact pressure distribution across the face of the indentor. Work by Hirst and Howse (1969) suggested that the stress distribution across a pyramidal indentor could be uniform providing that the Young's modulus to yield stress ratio of the material was high, and the indentor angle was acute. Nevertheless the model proposed by Lawn and Evans provided a basis which other authors referred to, even if their models were in contradiction.

It was Perrot (1977) that used these conclusions and stated that the expanding cavity model used in the Lawn and Evans model was not applicable as the 136 deg Vickers pyramidal indentor angle would not create an analogous uniform stress distribution. Also, the model was limited only to very brittle materials as it carried an assumption that the material would not exhibit plastic behaviour and therefore no material pile-up would occur. Lawn and Evans had also derived equations for the maximum load and flaw size required to initiate a median flaw. This was done by measuring the effect of loading on different size cracks in various materials. Large cracks above a critical size were made to extend stably with increasing load, and only critical intermediate size cracks grew unstably to form sub-surface median cracks at constant load. Perrot claimed that a calculated flaw size for median crack initiation in certain materials would mean that they would be easily detectable, and this was shown not to be so according to results by Hagan (1979). Perrot himself (1977) proposed that the stress field created by an axially symmetric obtuse indentor in a material with some plasticity would not first create median cracks which would grow into the radial/median formation, but the radial cracks would initiate
first. His analysis of the stress field predicted that the maximum tensile field occurred at the corners of the indentation and only in a shallow region within the plastic zone. This would explain the surface radial "Palmquist" cracks (Palmquist 1957) observed by various workers (Evans and Wilshaw 1976, Ogilvy et al 1977, Hogan and Swain 1978, Lankford and Davidson 1979).

Lankford (1981) agreed with Perrot's analysis of the stress field and theory that radial preceded the median cracks but this was the only important objection to the Lawn and Evans model. He proposed then that if the Lawn-Evans sub-surface maximum tensile stress was replaced with Perrot's near-surface maximum tensile stress the original model would be valid for Vickers indentations. Similar to the Lawn-Evans model the presence of fortuitous flaws were again assumed and these would be found by the tensile field in the plastic zone, this time in the near-surface. Thus Palmquist cracks would be formed from presumably the largest or "weakest" near-surface flaws (Figure 4.2a). Further loading would create a sub-surface penny-like crack formation (Figure 4.2b), which on unloading would spread together with the surface cracks to form the median/radial crack system (Figure 4.2c). This theory was lent support by studies into the effect of material surface finish on the radial crack initiation load (Evans and Wilshaw 1976, Haranoh 1982).

Chiang, Marshall and Evans (1982) followed this work with the most "complete" model up to now, which contradicted the conclusion on the position and nature of the near-surface stresses in the work carried out by Perrot and Lankford. An initial discrepancy lay in the prediction of material behaviour around an indentation site. Chiang et al revealed that materials subjected to large angle indentations, or materials of low Young's modulus, deform by radial compression which result in little material pile-up around the indentation site and a generally found hemispherical plastic zone. The claim that the Lawn-Evans model was limited as it didn't assume plasticity of the material was shown not to be so as even materials of high plasticity (ie brass) followed this behaviour. In terms of the indentation-induced stresses, Perrot predicted that the near-surface stresses were tensile and existed only in the plastic zone. However, various workers (Evans and Wilshaw 1976, Evans
Figure 4.2 The model proposed by Lankford (1981) for fracture initiation beneath a sharp (Vickers) indenter. In this model, radial crack initiation (Figure 4.2a) precedes median (Figure 4.2b). Loading (+) and unloading (−) cycles are as shown.
1979, Marshall et al (1979) observed that radial cracks terminated within the plastic zone. Also, radial cracks were seen to extend outside the plastic zone (Lankford 1981). This indicated that there existed a compressive stress within the plastic zone, and a wider stress field than Perrot had predicted.

Chiang, Marshall and Evans provided an analysis of the near-surface stresses under a pyramidal indentor which took account of the effect of the free surface by using a hemispherical cavity model. This had not been accounted for in the Lawn and Evans (1977), and Perrot (1977) models. Their results showed that regardless of the crack system ie (pyramidal or spherical indentor):

1) Maximum tension occurred at the plastic-elastic boundary.

2) The maximum tension decreased rapidly within the plastic zone which is highly compressive.

3) The maximum tension decreased monotonically as it entered the elastic zone.

Further information could also be specified for particular crack systems, where for radial cracking, the residual tensile surface stress approximates or exceeds the peak load tensile surface stresses (depending on the material and indentor size) (Figure 4.3a).

Also, for large indentor angles ie Vickers indentors, the geometric factors should cause radial and lateral cracking to be suppressed during loading, and this was confirmed in results obtained by Lawn et al (1980). For median cracks the peak load tensile stress beneath the indentor would always exceed the residual stress but would be less than the surface tensile stress.

These favourable results were then applied in the complex description of crack initiation, which up to then had not undergone a complete and rigorous analysis. The radial cracks were in fact found to initiate first and this was due to the combination of the high surface tensile field and the presence
Figure 4.3a Tangential surface stresses for radial fracture at both peak indenter load (upper and lower bounds) and indenter removal (residual), for two choices of relative plastic zone size (β). From Chiang, Marshall and Evans (1982)

Figure 4.3b Half-penny radial crack formation resulting solely from surface radial (Palmqvist) cracks. Loading (+) and unloading (−) cycles are as indicated
of surface flaws (Chiang et al 1982). On unloading the cracks would continue to grow into the elastic zone and also to the plastic zone where they were quickly terminated. However an interesting addition to the complex crack initiation problem was that Chiang et al (1982) claimed that if the surface finish is very good or surface compressive stresses are introduced, then radial crack formation may be suppressed and median cracks form on loading. Many brittle materials which exhibit microplasticity during indentation loading contain a near-field maximum tensile stress which initiates median cracks when the sub-surface flaw density is low, with the cracks finally growing to form surface radial cracks on unloading.

Summarising therefore, for a well developed Vickers median/radial crack system, where a load in excess of that required to produce radial cracking is used, there are three possible situations:

a) Radial cracks are first to nucleate from surface flaws at an early stage in the loading cycle, but are prevented from developing until the unloading cycle. As the loading cycle continues median cracks form at the plastic/elastic boundary, also from pre-existing flaws, and form sub-surface penny-like cracks immediately below the indentor. These grow on further loading and may engulf the radial before the unloading cycle commences.

b) Median cracks may form at a load not greatly in excess of that required to form the radial cracks. On unloading then, both crack systems grow simultaneously with the radial system confined to the near surface region. As unloading nears completion, the median cracks may merge with, or engulf, the radial cracks (Evans and Wilshaw 1976) resulting in a final crack pattern identical to the one formed in a).

c) If the load is insufficient to form a penny-like median crack but in excess of that necessary to initiate the radial system (Figure 4.3b), the surface radial cracks may grow on unloading, merging beneath the indentor tip to once again form the Vickers median/radial crack system.
4.3.3 Indentation Crack Propagation—Far Field Stresses

"Far-field" stresses influence the growth of an initiated crack and are more understood and less controversial than the near-field stresses.

Outside the contact zone the stresses in the material are elastic. The equations for the components of these elastic stresses about a point indentation in an isotropic material were first formulated by Boussinesq (1885). The stresses have the general form:

\[
s_{ij} = \left( \frac{P}{\pi R^2} \right) f_{ij}(\phi) \nu \quad \ldots \ldots \quad (4.8)
\]

where \( P \) the applied load, \( R \) and \( \phi \) are defined as in Figure 4.4 and \( \nu = \) Poisson's ratio for an isotropic material.

From these equations stress contours of the three principal normal stresses are determined (Figure 4.4). The \( \sigma_{11} \) and \( \sigma_{33} \) stresses both lie in planes of symmetry through the load axis with \( \sigma_{11} \) everywhere tensile and \( \sigma_{33} \) everywhere compressive. The \( \sigma_{22} \) "hoop" stress is tensile to within approximately 52° of the surface and compressive elsewhere.

The Boussinesq equations show that the stress field is related to the Poisson's ratio of the material. Lawn and Swain 1975 claimed that the Poisson's ratio greatly affected the maximum tensile stress which was found to disappear completely for \( \nu = 0.5 \). The indentation technique is best suited to brittle fracture and brittle materials lie in the range \( \nu = 0.2 \) to 0.5 (Kelly 1988). To obtain a stress field which will result in a well defined crack (a crack which is large in comparison to the indent and therefore is governed by far field stresses) the material should be to the lower end of this range. The ceramic material studied in this thesis has \( \nu = 0.268 \) (Dr J G Rider, private communication).

Thus for a material of low Poisson's ratio, once a crack is initiated immediately below the indentor tip, it will continue to propagate in the \( \sigma_{33} \) direction downwards into the material, while maintaining orthogonality with the \( \sigma_{11} \) and \( \sigma_{22} \) stresses.
Figure 4.4 Contours of principal normal stresses in the Boussinesq field, shown in plane containing load axis. Poisson's ratio taken as \( v = 0.25 \). Unit of stress is \( \rho_0 \), the mean contact pressure (After Lawn and Swain 1975)
It will also expand outwards in the $\sigma_{11}$ direction in order to remain orthogonal to the $\sigma_{22}$ tensile stress. This ultimately leads to the enclosed full-penny configuration (Figure 4.5).

This sideways expansion will, however, be limited by the compressive lobes of the hoop stress at angles of less than approximately $52^\circ$ to the surface.

Upon unloading, the material attempts to release its stored elastic energy. The plastic zone, being the most highly deformed region, undergoes the most severe contraction but is inhibited by the surrounding material. The residual tensile stress thus formed in the material surrounding the contact site drives the full-penny median vent to the equilibrium half-penny median-radial configuration maintaining near-orthogonality to the $\sigma_{11}$ radial tensile stress.

Alternatively, loading continues until the enclosed penny-like crack is able to break through the confining compressive stress lobes to once again form the median-radial configuration.

4.4 INDENTATION FRACTURE - FRACTURE TOUGHNESS AND STRESS INTENSITY FACTOR

To understand the effect of residual contact stresses on machining-induced cracks and therefore the strength of brittle materials it is helpful to consider the fracture toughness and stress intensity factor derived from an isolated elastic/plastic contact event. As discussed in Section 4.3 the description and analysis of residual crack patterns have mostly been carried out in studies using a sharp indentor, such as the Vickers pyramid indentor. Pertinent cracks form on median planes containing the axis of loading and the symmetry axes of the contact hardness impression; the characteristic radial traces provide a convenient record of crack growth. These radial crack patterns can be used to determine the fracture toughness $K_c$, of a material (Anstis et al 1981), where in the analysis the complex elastic/plastic field is considered in two separate "elastic" and "residuals" terms, and the residual component has a dominating role in the final size of the radial crack (Marshall and Lawn 1979, Lawn et al 1980).
Figure 4.5 Enclosed "full-penny" median vent formation beneath a sharp indenter as loading progresses. Radial vent formation (usually) occurs upon indentor removal but may also be formed during loading.
Figure (4.6a) shows a schematic diagram of the indentation deformation/fracture system produced by a peak load $P$. By simple dimensional analysis it can be shown that the hardness $H$ and toughness $K_c$ of the material are related to the characteristic parameters $c$ and $a$ of the penny-like radial/median cracks and hardness impression via the following equations:

$$H = \frac{P}{\alpha_0 a^2}$$

$$K_c = \frac{P}{\beta_0 c^{3/2}}$$

where $\alpha_0$ and $\beta_0$ are constants $\alpha_0 = 2$ for Vickers geometry and $\beta_0$ corresponds to a complex geometrical factor for penny-like systems, where interaction effects due to the free specimen surface, and multiple-plane crack configuration are incorporated. The term is calculated by experimental calibration.

In addition to the median/radial crack system a lateral crack system can also be generated (Figure 4.6b). These cracks spread outward from the deformation zone beneath the indentation surface, and may interact with the radial crack system. In severely loaded specimens they turn upward to intersect the surface thereby causing severe disruption of the pattern by chipping. Therefore hardness and fracture toughness measurements are taken within a workable range of loads to avoid this problem.

The radial crack evolution is understood by considering the elastic/plastic field as two superposable elastic and residual components which contribute to the driving force on the crack system (Lawn and Wilshaw 1975, Marshall and Lawn 1979). At the indentation surface the elastic component is compressive and the residual component tensile. Thus radial cracks grow to their final lengths as the indentor is unloaded. The residual field is therefore considered primarily responsible for expanding the crack system into its penny-like configuration.
Figure 4.6a  Schematic of Vickers indentation fracture system produced by a peak load $P$, showing characteristic penny-like radial/median crack and hardness impression, denoted by $c$ and $a$ respectively.

Figure 4.6b  Schematic, cross-section showing deformation zone and lateral cracks.
The residual field has been evaluated to a greater depth by Lawn et al (1980), in terms of an outward-acting pressure at the boundary of the plastic zone, which occupies an almost hemispherical volume of radius $b$. For sufficiently developed cracks ($c \gg b$), the penny cracks can be considered to be "centre loaded" at the deformation zone in a point force located at the crack centre. Under this condition the driving force is characterised by the stress intensity factor for the radial crack:

$$K_r = \frac{XrP}{c^{3/2}}$$

where $Xr$ is a constant. $Xr$ is related to the manner in which the volume of plastic impression is accommodated by the surrounding elastic matrix, and therefore is dependent on the elastic modulus $E$ and hardness $H$ of the material. Lawn et al carried out a detailed treatment of the expanding cavity model, using it to derive the expression for $Xr$:

$$Xr = \frac{f^R}{\nu} \left[ \frac{E}{H} \right]^{\nu}$$

Therefore the stress intensity factor is given by

$$K_r = \frac{f^R}{\nu} \left[ \frac{E}{H} \right]^{\nu} \frac{P}{c^{3/2}}$$

where $f^R$ is a dimensionless constant dependent only on the indenter geometry and is an intrinsic stress/strain response of the material. It is determined experimentally. Assuming mechanical equilibrium ($K = K_e + K_r = K_c$) is achieved after unloading, such that the radial cracks remain stable, then $K_e = 0$ and $K_r = K_c$. Denoting $c = c_o$ as the half-penny crack dimension after indentation, then equation 4.13 gives an expression for fracture toughness $K_c$:

* $K_e$ is the elastic component.
where $P$ is the peak load.
The validity of this equation has been tested with Vickers indentation in a wide range of ceramic materials (Anstis et al 1981).

4.5 Indentation Crack Response to Applied Loading

In the correlation of flaw dimensions with failure strengths, conventional Griffith concepts have normally been used. However, indentation fracture studies (Marshall and Lawn 1979, 1980) have shown that there are substantial differences from ideal Griffith flaws in the mechanics of failure generated by indentation cracks. It is the influence of the residual stress field associated with the elastic-plastic damage that causes the two crack types to behave differently. The residual field assumes a dominant role not only in the evolution of indentation cracks, but also in the response of the cracks to an applied loading. Instead of failure occurring at a critical applied stress, without precursor extension (as in the case for Griffith cracks), the instability of indentation flaws is achieved after a region of stable, equilibrium growth. The failure stress is independent of initial crack length and is appreciably lower than the strength associated with stress-free cracks of equivalent initial dimensions.

Consider the Vickers-induced radial crack system subjected to a normal tensile stress $\sigma_a$ (Figure 4.7a). The stress intensity factor appropriate to this tensile loading has the standard form (Lawn and Wilshaw 1975):

$$K_a = \sigma_a (\pi \Omega c)^{1/2}$$

where $\Omega$ is a crack geometry parameter. According to conventional strength theory failure will occur spontaneously from the starting flaw at some critical stress level, provided a state of
Figure 4.7a Schematic of Vickers-produced radial/median crack system under tensile loading from applied stress $\sigma_a$ with contribution from residual stress field

Figure 4.7b Schematic of crack configurations generated by linear damage processes (row of indentations, scratching or machining) and the crack front at instability.
mechanical equilibrium is maintained throughout tensile loading. At the critical conditions, writing \( \sigma_a = \sigma_0, \ K_a = K_c \), equation 4.15 gives the strength relationship:

\[
\sigma_0 = \frac{K_c}{(\pi \Omega c_0)^{1/2}}
\]

.......

(4.16)

where \( c_0 \) is the radial crack size prior to application of the applied stress. The expression tells us that the strength is a function solely of the original crack configuration. However, there is another driving force acting during the tensile loading, and this is the residual term (equation 4.12). The net stress intensity factor is therefore:

\[
K = K_a + K_z - \sigma_a (\pi \Omega c)^{1/2} + \chi_z \frac{P}{c^{3/2}} \quad (c > c_0)
\]

.......

(4.17)

For growth under equilibrium conditions the critical stress intensity factor, \( K_c = K_a + K_z \). The failure condition is solved by putting the applied stress as a function of crack size

\[
\sigma_a = \left[ \frac{K_c}{(\pi \Omega c)^{1/2}} \right] \left[ 1 - \frac{\chi_z P}{K_c c^{3/2}} \right]
\]

.......

(4.18)

The failure condition is defined by the maxima:

\[
\sigma_m = \left[ \frac{27}{256} \frac{K_c^4}{(\chi_z \pi \Omega)^{3/2}} \right]^{1/3} P^{-1/3}
\]

.......

(4.19)

thus
Summarising, the indentation crack undergoes a stage of precursor stable growth from $c_o$ to $c_m$, in attaining an instability configuration at $\sigma_o = \sigma_m$, which now defines the as-indented strength (Chantikul et al 1981). This behaviour contrasts with the response of ideal stress-free cracks ($\chi_r = 0$, $c = c_o$ in equation 4.18) where crack instability is achieved at a critical applied stress level without precursor extension. In situ measurements of the surface traces of Vickers cracks during failure testing have demonstrated the existence of stable crack extension according to equation 4.18 in a wide variety of ceramic materials (Marshall et al 1979b, Lawn et al 1981, Marshall 1981, Cook et al 1982). Also strength degradation measurements have confirmed the predicted dependence of strength on materials properties and contact load (equation 4.19) (Chantikul et al 1981).

4.6 MACHINING-INDUCED DEFORMATION FRACTURE AND RESPONSE TO APPLIED LOADING

The deformation and fracture generated by individual particle contacts during machining are expected to resemble the damage associated with sharp diamond indentations. The micro mechanics of crack formation and propagation in machining damage might therefore be expected to exhibit analogous residual stress dominated effects.

This prediction has been demonstrated by the reduction in strength of ceramic surfaces with single-point machining damage (Kirchner and Isaacson 1982 and 1983). The analysis shows that residual stresses caused by the linear deformation - fracture configuration provide a similar crack response under applied load to indentation fracture analysis. The region of stable precursor
crack growth though, is more extensive $c_m/c_o = 4$ than for axisymmetric penetration, $c_m/c_o = 2.5$. The analysis is only applicable to cracks generated by the penetration of a wedge indentor.

However, experiments have shown that strength-degrading cracks form on median planes in a series of semi-circular or semi-elliptical sub-surface cracks beneath the most severe machining grooves (Rice and Mecholsky 1979, Marshall et al 1983) (Figure 4.7b). This suggests that the loading during machining may resemble more closely the axisymmetric indentation. It is expected that such geometrical deviations from linear geometry would reduce the $c_m/c_o$ ratio.

The factor that probably causes machining cracks to behave differently from the idealised indentation cracks (linear or axisymmetric) is the overlap and interaction of residual stresses from neighbouring damage sites. Similar to an indentation site (Lawn et al 1980), or an isolated grinding groove (Figure 4.8a) the volume of a machining groove is accommodated by the surrounding material. The subsequent plastic zone has an outward-acting pressure at its boundary which causes a residual stress, and tension on median planes beneath the zone. The cumulative effect of many neighbouring damage sites of similar depths, with a high degree of overlap in the residual fields, is the formation of a uniform thin layer of residual compression with an underlying residual tension.

However, the strength-controlling damage extends to a greater depth than the damage in neighbouring regions (Figure 4.8b) and is only part counter balanced by the layer of residual compression. The layer therefore reduces, but does not eliminate the localised tensile crack-opening force (Cook et al 1981, Kirchner and Isaacson 1982 and 1983, Marshall et al 1983, Marshall 1984). Under the influence of an applied tensile load, together with the effect of the residual stresses, the semi-elliptical cracks undergo stable growth prior to failure (Marshall et al 1983). Crack extension begins at certain preferred locations probably due to high local residual stresses, and is then followed by coalescence to form a single semi-elliptical crack, which continues to propagate stably until
Figure 4.8a Schematic of isolated grinding groove or indentation: residual pressure $p$ at the boundary of the plastic zone creates compression adjacent to the zone and tension on median planes beneath the zone.

Figure 4.8b Machined surface: compression layer due to overlap of neighbouring residual fields competes with the upper portion of the outward-acting pressure from the strength controlling groove, thus reducing the residual crack-opening force, $P_r$. 
failure occurs. Using an analysis where the crack configuration at the failure point is given by $C_m = (C_s C_d)^{1/2}$ (Bansal 1976) (Figure 4.7b), Marshall et al. (1983) measured the extent of stable crack growth during failure for a series of machined and indented HPSN ceramic surfaces. The ratio $C_m/C_o = 5$ was consistent for all the combinations, and is high compared with $C_m/C_o = 2.5$ for isolated indentation damage and $C_m/C_o = 4$ for the linear crack configuration. This is due to an extensive stable expansion of the crack parallel to the surface. Using the failure strengths and the corresponding measured crack dimensions at failure the following relation was found:

$$\sigma = \frac{3.9}{C_m^{1/4}} \text{ MPa}$$

... ... (4.22)

The equation is in the same form as the Griffith strength equation, but contains the crack length at failure, rather than the original crack length $C_o$. These results are however empirical and cannot be correlated to the micro-mechanics of machining-induced crack response to applied loading.

4.7 THE RESIDUAL COMPRESSIVE LAYER - DAMAGE RESISTANCE AND DELAMINATION

There is a competing process between the machining-induced compressive layer and the residual localised tension at a strength-controlling flaw. An enhancement of the compressive layer relative to the tensile crack-opening force would increase the strength, and this could be done by modifying machining procedures. The compressive layer can also improve in-service strength degradation by mechanical contact, as shown in indentation studies (Cook et al. 1981, Marshall et al. 1983).

However, the competing process between the compressive and tensile fields may also be damaging, and can be understood as follows. The nature of the stresses may be described by considering the deformed layer as containing a row of edge dislocations (Badrick et al. 1979). The opening up of wedges in the surface are "filled" by a greater volume of material. An
expansion of the surface therefore causes the formation of a residual compressive stress with an underlying compensating tensile stress. At the plastic/elastic boundary there is a singularity, where a physical boundary exists between the deformed and undeformed region. This is an area of weakness where a crack may initiate, and then propagate due to the outward expansion and arcing of the deformed layer. This problem of delamination is a common problem in machined materials that contain resultant longer range biaxial fields.

4.8 STATISTICS OF FRACTURE STRENGTH

4.8.1 Fracture Strength

In general terms the fracture strength of ceramics is related in some way to the flaw size, $a$, via the relationship:

$$\sigma \propto \frac{K_{rc}}{a^{\frac{1}{n}}}$$

(4.23)


The flaws may be formed during fabrication or by contact damage after fabrication. Once cracks are present they are not blunted by the action of plastic flow as in non-brittle materials. The exact response of flaws to applied loading will differ according to the flaw types and specific local conditions (Marshall et al 1983, Evans 1984, Govila 1988). Testing a batch of nominally identical ceramic specimens will generate a distribution of fracture strength values as the flaw size varies in each specimen. The sample with the largest critical flaw will be the weakest.

4.8.2 The Weibull Distribution

An empirical analysis of fracture strength data can be carried out using Weibull statistics; although no single tensile strength can be attributed to the material, there does exist a definite probability that a given sample will have a given strength. The failure probability $P_f(V_o)$ is the fraction of nominally identical samples of volume $V_o$ that fail loading to a
tensile stress $\sigma$:

$$P_f(V_o) = 1 - \exp\left[\left(\frac{\sigma}{\sigma_o}\right)^m\right]$$

... ... ... (4.24)

where $\sigma_o$ and $m$ are constants. Setting $\sigma = \sigma_o$ then $P_f(V_o) = 1 - 1/e = 0.63$. Thus $\sigma_o$ is the stress at which 63% of the samples will fail (Figure 4.9a). The Weibull modulus $m$ describes the degree of variability in strength, with a higher modulus signifying a more well-defined tensile strength. For ceramics $m$ is generally around 10. A material with a value of 20 does not have a higher strength, but a more uniform flaw size. To evaluate $m$ natural logarithms are taken of equation 4.24 giving:

$$\ln\left[\ln\left(\frac{1}{1-P_f(V_o)}\right)\right] = m \ln \frac{\sigma}{\sigma_o}$$

... ... ... (4.25)

A plot of

$$\ln\left[\ln\left(\frac{1}{1-P_f(V_o)}\right)\right]$$

versus $\ln \frac{\sigma}{\sigma_o}$ or direct use of the Weibull probability axes gives a straight line of gradient $m$ (Figure 4.9b)(Ashby and Jones 1986).

4.8.3 Rank Order

Dividing a batch of specimens into groups and testing each group at a given stress is time consuming and not economical. A better method of achieving data is to apply an increasing stress until failure occurs, thereby allowing the specimens to be ranked in order of increasing fracture strength. However, there is a difficulty in assigning a $P_f$ value to each rank position as the failure of one specimen at a particular stress does not mean that the rest of the specimens would have passed that level of stress. Statistical considerations provide an expression for the most probable value of the failure probability.
Figure 4.9a The Weibull distribution function, where the probability of survival $P_s = 1 - P_f$

Figure 4.9b Survival probabilities $P_s = 1 - P_f$ for three different materials plotted on Weibull Probability Axes
of the $i^{th}$ specimen in a group of $n$:

$$P_f(V_i) = \left[ \frac{i-0.3}{n+0.4} \right]$$

... ... ... (4.26)

4.8.4 Volume Dependence

As the measured strength is dependent on the volume being stressed, a volume $V_o$ has been assigned to the probability $P_f$. The larger the specimen the more likely it is to contain a larger flaw and hence the lower its strength is likely to be.

The probability of a sample failing a stress $\sigma$ is $P_f(V_o)$. The probability of $x$ samples failing is $[P_f(V_o)]^x$. If these $x$ samples were stuck together to make a larger sample of volume $V$ ($= x \times V_o$) then its probability of failure would still be $[P_f(V_o)]$. Thus:

$$P_f(V) = [P_f(V_o)]^x = [P_f(V_o)]^{x/V_o}$$

and

$$\ln[P_f(V)] = \frac{V}{V_o} \ln[P_f(V_o)]$$

... ... ... (4.27)

or

$$P_f(V) = \exp\left[ \frac{V}{V_o} \ln[P_f(V_o)] \right]$$

... ... ... (4.28)

Substituting

$$\ln [1-P_f(V_o)] = -\left( \frac{\sigma}{\sigma_o} \right)^m$$

from equation 4.24 gives

$$P_f(V) = 1- \exp \left[ -\frac{V}{V_o} \left( \frac{\sigma}{\sigma_o} \right)^m \right]$$

... ... ... (4.30)
4.9 SINGLE POINT SCRATCH TESTS

The nature of machining damage is complex with multi particle contact events creating surface and sub-surface damage in the form of chipping, grain pull-out, cracks and plastic deformation. To understand the material removal processes involved it is convenient to study the deformation caused by a simplified condition of machining damage - single point scratching with a Vickers pyramid diamond indenter.

The stress trajectories and principal tensile stresses for a sliding sphere were analysed by Hamilton and Goodman (1966), Lawn (1967) thus providing a solution for the elastic stress distribution or Hertzian stress field. Swain (1978) made observations on microcracking about Vickers pyramid diamond scratches in a number of brittle solids, and considered the advent of elastic fields, as well as plastic deformation and residual stress in the analysis. He found that the nature of cracking is similar to that occurring about a quasi-static point indenter, where directly beneath the indenter a well-defined median crack is formed. Behind the indenter small partial hertzian cracks were observed to develop within the track, and sub-surface lateral cracking initiated from the plastically deformed zone. The residual stress was claimed to be the driving force for the growth of lateral cracks (See Section 4.3). The asymmetric nature of the indentation stress field due to tangential tractions had already been considered in three dimensions by Evans (1976) where the assumption was made that the lateral cracks occurred at the head of the scratching point.

Rice and Mehlolksky (1979) studied the nature of machining flaws from multi-point diamond grinding in glass, single crystal and polycrystalline materials based on the fractographic analysis of the flaws as origins of failure in 3 point flexural rupture tests. Anisotropy in strength due to the direction of grinding relative to the tensile axis was found to be due to the presence of two populations of flaws of different form, one set perpendicular and the other set parallel to the grinding direction. The perpendicular flaws typically approached a semi-circular periphery form while the parallel flaws were found to be typically more elongated and often larger than the former (See
Section 4.6), thus giving rise to the lower strengths where the applied tensile stress was perpendicular to the grinding direction.

Indentation fracture studies by Marshall et al (1979), Marshall and Lawn (1980) showed that substantial differences from ideal Griffith flaws exist in the mechanics of failure from cracks generated by sharp particle contact during a single point or multi point machining process. The difference in behaviour results from the residual stress field associated with elastic/plastic damage, where the residual field plays a dominant role in the evolution of indentation cracks and in the subsequent response of the cracks to an applied loading. Instead of failure occurring at a critical applied stress without precursor crack extension (as is the case for Griffith cracks) the instability of indentation flaws is achieved after a region of stable, equilibrium crack growth.

The nature of residual stresses in hot-pressed silicon nitride grooved by single point grinding and their effect on material strengths was studied by Kirchner and Isaacson (1982). Hot pressed silicon nitride specimens were grooved by single diamond points with varying degrees of flatness mounted on a wheel rotating at varying speeds. The depths of damage were measured and used to calculate the theoretical strengths based on conventional Griffith concepts. For sharp diamonds and in some cases diamonds with flat tips, the measured strengths were found to be less than the theoretical strengths and the differences were attributed to the presence of residual loads acting to wedge open the cracks (See Section 4.6). High wheel speeds using the sharp diamonds resulted in an increase in the differences between theoretical (Griffith-based) and measured strengths, particularly for smaller flaws. This indicated the presence of higher residual loads due to higher levels of plastic deformation. At the same time, at a particular wheel speed and crack depth, the remaining strengths were found to increase with increasing flatness of the diamond points. Although sub-critical crack growth had already been observed in hot pressed silicon nitride (Gulden and Metcalfe 1976) the effect was considered to be insignificant in this case, also following other analyses of residual stresses from static indentations (Petrovic and Jacobsen
Further work by Kirchner and Isaacson (1983) was carried out on steatite, hot-pressed silicon nitride, glass and hot-pressed silicon carbide. Measured strengths for glass and HP SiC fell close or above Griffith-based theoretical values, where residual stresses were interpreted to be low due to extensive crushing and a large fraction of the residual stress immediately being relieved by lateral cracking or other similar mechanisms when the diamond point passed on. Furthermore, the force ratios were found to be lower than for stealite and HP Si₃N₄ indicating that the low resistance to horizontal motion of the diamond point was due to the fact that the materials crush relatively easily; the material that would otherwise be subject to plastic deformation was crushed and swept out of the groove instead.

Measured strengths of steatite, and again HP Si₃N₄, both fell below Griffith-based theoretical values. The force ratios (horizontal to vertical) were relatively high indicating a substantial resistance to horizontal motion of the diamond point, and this was attributed to plastic flow (rather than crushing) in the interface with resulting high residual stresses.

Further work by Kirchner (1984a,b) concluded that the damage penetration at low vertical loads was approximately proportional to the load, whereas at high load, the load dependence of the crack length was much greater.

Imanaka et al (1972) had directly observed chip removal processes during the grinding of glass-ceramics and several types of oxide ceramic by a micro-flash technique. The chips were found to be generally of a fragment type and were distinguished from those of ductile materials. Chip removal occurred via two processes; either by direct removal by the diamond point or by splintering after the passing of the diamond point.

Zhang et al (1988) carried out scratching experiments on hot pressed alumina using single point diamonds of conical shape with varying tip included angles and nose radii. For diamonds of nose radius 1.1μm to 1.9μm and a depth of cut less than 1μm little material removal was found although microcrack cluster formation beneath the groove was observed. With an increasing depth of cut
the groove surface area exhibited macroscopic plastic deformation through to scale-like cracking, followed by cracking or chipping, accompanied by a sub-surface median crack.
CHAPTER 5. EXPERIMENTAL PROCEDURE

SUMMARY
This chapter describes how as-hipped ceramic billets were prepared and machined into 3 point flexural rupture test bars for investigation and shows how some very innovative techniques were used to study machining damage in the component surfaces.

5.1 DIAMOND MACHINING THE CERAMIC

The procedure for shaping (diamond cutting and grinding) as-hipped ceramic billets and peripheral diamond machining the resultant 3 point flexural rupture test bars is given. A method which employed a Talystep stylus instrument for the measurement of the coarse, intermediate and fine levels of surface finish to be studied is outlined.

5.2 CROSS-SECTION EXAMINATION OF COARSE MACHINED SURFACE

The importance of minimising specimen preparation defects in the study of machining damage is introduced. To study the surface and sub-surface mechanical damage in the machined bars a potentially revealing but extremely difficult technique of producing sandwich cross-sections of the machined surface was employed. The various delicate stages of bonding, cutting, coring and mechanical polishing are detailed including preparation for optical, scanning and transmission electron microscopy.

5.3 METHODS OF OBSERVATION

A macro view of the machined surfaces was carried out with optical and scanning electron microscopy. A cross-section examination of median cracks was made possible by an as yet undocumented technique where features were highlighted by a "penumbra" or diffuse, oblique illumination caused by the closing down of the field stop in the optical microscope. An examination of the residual compressive layer was also carried out using an inventive technique employing this time transmission electron microscopy on the sandwich cross-section. The position and
extent of mechanical damage in the sub-surface was detected via distortion of X-ray microdiffraction spots.

5.4 THE EFFECTS OF MECHANICALLY POLISHING THE MACHINED SURFACES AND A SURFACE CONTAINING AN INDENTATION

A description is given on how the mechanical polishing of a machined or indented surface causes interaction effects which allow an analysis of the machining damage to be gained from a different perspective.

5.5 EFFECT OF INDENTING A COARSE MACHINED SURFACE

This shows how a machined surface should be indented in order to gain information on the machining-induced mechanical damage.

5.6 THREE POINT FLEXURAL RUPTURE TESTS

The preparation of machined bars and the procedure in carrying out 3 point fracture tests is given.

5.7 SINGLE POINT SCRATCH TESTS

Preparation and scratching techniques are discussed, together with the method of analysing resultant material debris, and producing cross-section views of the scratched surface.
CHAPTER 5  EXPERIMENTAL PROCEDURE

5.1 DIAMOND MACHINING THE CERAMIC

In April 1988, seven months after a previous HIP run at ASEA, a new batch of material was fabricated. One of the five billets was examined and a short analysis was made of the processing flaws due to the urgency in directly studying the effects of diamond machining. A three dimensional white cellular phase structure similar to the one present in the batch of material from the August 1987 HIP run was found in all five billets, (See Section 3.16, Figure 3.18). The cell size $= 2.1\text{mm} (\pm \sigma = 0.1\text{mm})$. Metallic agglomerates ranging from $3\mu\text{m}$-$14\mu\text{m}$, with a few reaching $25\mu\text{m}$ were also detected (See Section 3.2.2.4, Figure 3.28).

5.1.1. Preparation of Billets for Surface Machining

Bars of dimensions $3\text{mm} \times 3\text{mm}$ cross-section and $50\text{mm}$ length were diamond cut and ground to size from the five as-hipped billets at P S Marsden Precision Engineering Limited.

The facilities at this company include two Jones and Shipman L540 horizontal spindle grinding machines (Figure 5.1). The spindle speeds are a set $2650 \text{ r.p.m.}$, the table speed can be set at $4\text{cms}^{-1}$ or $20\text{cms}^{-1}$, and the depth of cut handwheel is operated manually to an accuracy of $1\mu\text{m}$. The work table cross-feed also has the same accuracy. The grinding wheels used for ceramics are Cape Diamond resin bonded diamond wheels; a grit size number and diamond particle equivalent is given in Table 5.1.
Figure 5.1  Jones-Shipman grinding machine
### TABLE 5.1: DIAMOND WHEEL PARTICLE SIZES

<table>
<thead>
<tr>
<th>Cape Diamond code number</th>
<th>BS 410 equivalent</th>
<th>Diamond Particle size µm</th>
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<tbody>
<tr>
<td>36</td>
<td>36/44</td>
<td>425/350</td>
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<tr>
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<td>44/52</td>
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</table>

The diamond grinding wheels are dressed each time a new component is ground (or a new wheel is mounted). This is carried out with a SiC vitrified bond wheel crusher which can be mounted on the machine work table. All grinding operations are carried out under flood cooled conditions using a water soluble oil, Cimcool MB 301, and Weir-type coolant filters (Figure 5.2). The spindle runs on a ball bearing shaft and dynamic wheel balance is
Figure 5.2 Flood cooled bars being transversely ground
maintained with counter balancing weights. However, machine vibration can occur due to local contact conditions between the wheel and the work piece surface, and resultant "chatter marks" are avoided by dressing the wheel during the grinding operation. As finished machined surfaces of 0.05µm CLA roughness were required for the project, the older grinding machine was reserved only for rough grinding operations.

Initial shaping of a 22mm diameter, 10cm long billet required the removal of the spherical end which was carried out using a 20cm diameter, 1.8mm thick, steel-backed 150 grit diamond cutting wheel. The remaining cylinder was then clamped in a vice on the machine work table and plunge ground into a rectangular block to within 25µm of size, using an 18cm diameter 150 grit resin bonded diamond wheel, (Figure 5.3a). The remaining 25µm layer was removed by rough machining with the same diamond grit size wheel and a table speed of 20cms\(^{-1}\), and this sometimes caused chipping on the block edges.

The block was then cut into 3.5mm high slabs and then into 3.5mm wide bars with the 150 grit diamond cutting wheel and a table speed of 20cms\(^{-1}\) (Figure 5.3b, 5.3c).

All cuts were made half way down with the block then being turned over for the final cut, as this would help to avoid the formation of burrs. The sides of 3.5mm wide bars were then ground to size with the grinding wheel. The surface which eventually would undergo tensile stress in a three point beam test was not machined last. Instead the surface underwent the machining schedule as described below in Section 5.1.2, and then the back face was finally machined to 3.0mm size. Therefore any chipping occurring on the back face would not present any problems as this surface would be in compression during a flexural rupture test. Finally, the edges of all machined surfaces were chamfered to avoid the occurrence of edge induced failures. This was carried out by longitudinally machining at an angle of 45° with a 150 grit cup wheel and finishing with a 400 grit cup wheel.

5.1.2 Surface Machining of Flexural Rupture Test Bars

5.1.2.1 Coarse Machining

Initial rough grinding was carried out with an 18cm diameter
Figure 5.3a Cross-section schematic view of the plunge grinding process

Figure 5.3b Schematic cross-section lateral and 3-d view of diamond cutting

Figure 5.3c Schematic cross-section and 3-d view of bar formation
150 grit resin bonded diamond wheel and a table speed of 20 cms\(^{-1}\). The bars were ground to 200\(\mu\)m of size using 10\(\mu\)m depths of cut, each time wound on manually by hand, producing a surface finish of around 0.5\(\mu\)m CLA roughness. This rough grinding procedure would occasionally cause chipping on the bar edges.

The machining procedure employed a 350 grit wheel taking the bars from: 200\(\mu\)m to 125\(\mu\)m of size with 10\(\mu\)m depths of cut at a table speed of 20 cms\(^{-1}\), 125\(\mu\)m to 25\(\mu\)m of size with 10\(\mu\)m depths of cut at a slower table speed of 4 cms\(^{-1}\), with the final 25\(\mu\)m being removed in 5\(\mu\)m depths of cut at a table speed of 4 cms\(^{-1}\). This procedure would leave the surface with about a 0.4\(\mu\)m CLA roughness finish. Bars were ground using the same schedule in longitudinal and transverse directions for flexural rupture test objectives.

5.1.2.2 Intermediate and Fine Machining

For intermediate and fine machining 400 and 600 grit resin bonded diamond peripheral grinding wheels were used respectively, with a table speed of 4 cm s\(^{-1}\). By setting a 5\(\mu\)m depth of cut and leaving the bars to pass backwards and forwards under the wheel, surfaces of 0.1\(\mu\)m and 0.05\(\mu\)m would be produced.

However, the machine operator has found that when a standard machining procedure is used, various types of ceramics of different hardness yield different qualities of surface finish. As the objective was to achieve specified surface finishes of 0.1\(\mu\)m and 0.05\(\mu\)m CLA roughness, when the April 1988 sinter hipped material did not yield the required result, the operator departed from the schedule. Therefore in some cases 220 grit or 400 grit bell grinding wheels were used, which had the equivalent effect as 400 and 600 grit peripheral diamond grinding wheels respectively. Consequently this did not allow a valuable analysis of the bars with these surface finishes. After 2 years and 8 months of the project, bars with suitably machined surfaces were supplied.

The diamond machining operator at P S Marden Precision Engineering Limited has found that many silicon nitride ceramics contain what he describes as a "mottled effect" on the surface, and that these areas contain an enhanced pull-out as a result of
machining (see Section 3.3.1.1).

5.1.3 Measurement of Surface Finish.

At P S Marsden LTD the surface finish was measured with a Taylor-Hobson Surtronic 2 instrument (Figure 5.4). An arm with a 4mm diameter ball and stylus tip at the end is passed in a backwards and forwards motion (normal to the grinding direction) on the surface. The surface roughness is translated into a voltage signal which is read by an indicator needle on a calibrated scale. At the University of Surrey the surface roughness was recorded with a Talystep instrument, which uses a stylus in the form of a truncated pyramidal tip of approximately 0.1μm x 2.5μm maximum value. The stylus is traversed over the specimen surface with the 0.1μm radius in the direction of traverse, and the vertical movement is amplified electronically and recorded graphically, producing a surface profile. From the profiles an analysis can made of the centre line average roughness, (Thomas 1982) (full details of Talystep and analysis methods in Appendix B).

5.2 CROSS-SECTION EXAMINATION OF COARSE MACHINED SURFACE

As discussed in Chapter 1, in the study of machining damage, it is important to minimise the introduction of defects during the specimen preparation stage. Unlike metals, ceramics cannot be etched. The only method for preliminary preparation is mechanical polishing, a difficult technique with brittle ceramics. A discussion of specimen preparation techniques is given in Appendix B.

To reveal sub-surface damage caused by diamond machining, cross-sections of the bars were prepared for microscope examination. However, to maintain the integrity of the free and sub-surface, a method of protection during specimen preparation was needed. A technique of producing a sandwich cross-section held together by an adhesive was therefore employed. It is a variant of an existing technique used on semiconductor materials.
Figure 5.4 Taylor-Hobson Surtronic 2 measuring transverse ground bars
5.2.1 Specimen Preparation

5.2.1.1 Bonding

A bar was diamond cut in two 15 mm pieces which were cleaned and de-greased in an ultrasonic bath with a) acetone b) trichloroethylene c) methanol at 50°C. For the adhesion of the two machined surfaces an epoxy adhesive was used as this does not dissolve in acetone; later specimen preparation would involve bonding with cyanoacrylate adhesive and unbonding in acetone. The epoxy part was mixed evenly with the hardener in a ratio of 1:1. A low amount of hardener was used as this would allow longer and therefore stronger polymer chains to form in the adhesive, even though curing time was greatly lengthened. The two machined surfaces were evenly spread with adhesive and were brought together. The surfaces were moved under pressure to evenly spread the adhesive and then a clamp was secured with the jaws as parallel as possible this being checked with a grid chart placed underneath. The sandwich was left to cure for 24 hours at room temperature.

5.2.1.2 Diamond Cutting Thin Slices

The bar sandwich was bonded on a Bakelite holder using cyanoacrylate adhesive, with the machined surface normal to the Bakelite surface. This would ensure that during diamond cutting the interface would be normal to the direction of feed and would not undergo any excess pressure (Figure 5.5a). Cyanoacrylate adhesive was set on the sides of the sandwich as well so that the slice about to be cut would not only have the support of the adhesive underneath but at the sides as well, thus avoiding the formation of burrs. Slices were cut on a Struers Accutom Cut-Off Machine, with a high concentration continuous rim 350 grit resin bonded diamond cutting wheel of 75mm diameter and 0.15mm thickness. A slow feed rate of approximately 0.15 – 0.25mm/min and a wheel speed of 1000rpm were used, with a fast alcohol-based lubricant drip rate (Full details of the Accutom machine are in Appendix B).

Slices were cut in a range of thicknesses from 200μm to 1500μm and it was found that specimens (400μm broke as a result of cutting. The slices were unbonded in acetone at 50°C.

5.2.1.3 Disc Coring

For the preparation of 3 mm diameter specimens to be used on
Figure 5.5a Diamond cutting the bar sandwich normal to the interface. The two machined surfaces are facing each other. See also Figure 5.7a for the shape of a single bar with chamfered edges.

Figure 5.5b Disc coring the sandwich cross-section slice to produce a 3mm diameter disc.
5.2.1.4 Surface Preparation For Optical and Scanning Electron Microscopy

Surface preparation was carried out using a VCR 500 Dimpler machine (details of use in Appendix B). A 3mm disc was first lapped with a 6µm diamond grit to remove at least 250µm from the as-diamond cut surface. This ensured that any mechanical damage caused by the diamond cutting was removed. 6µm of material were then removed with a 1µm lap, and finally the soft pad wheel with a 1µm diamond slurry was used to polish the surface. All stages were carried out with a wheel speed of 40° and a weight of 40gm.

5.2.1.5 Cross-Sectional Specimen Thinning for TEM

One side of a 3mm diameter disc was prepared as detailed in Section 5.2.1.4 above. An amount of material was removed so that the prepared surface was at the centre position of the original thickness. This ensured that the mechanical damage caused by the previous diamond cutting stage was removed. The specimen was then turned over and lapped with a 6µm diamond grit slurry to a thickness of approximately 150µm. Dimpling to a central thickness of around 20µm would then leave a small flat surface along the rim, allowing easier future specimen handling. The specimen was first dimpled to a central thickness of approximately 25µm with a 6µm diamond grit slurry, and then further dimpled with a 1µm diamond grit slurry to a central thickness of about 20µm. This was done with a wheel speed of 40° and a weight of 40gm.

Argon Beam Thinning:

Final thinning to electron transparency was carried out in an Ion Tech Ltd FAB Thinner, which uses two beams of energised Argon atoms. The ceramic/epoxy interface is the area of interest and so to prevent preferential thinning of the epoxy adhesive, two small brass rods were positioned on the tantalum plates holding the specimen, so that the interface would be shielded when the beams were parallel with it. A supply voltage of 5kV was used with a beam angle of 12°. A terminator gun which uses Argon ions was set at a sensitivity of 30µA, roughly equal to the
Figure 5.6  3mm diameter disc with interface slightly off-centre
ion gun currents.

5.2.1.6 Back-Thinning for Transmission Electron Microscopy
Background
Considerable time and effort was spent on trying to produce a
cross-sectional TEM specimen of the intermediate and fine
machined surfaces. However after trying various epoxy adhesives
and constituent combinations of hardener to epoxy fluid, the
machined surface/epoxy adhesive/machined surface interface would
not survive Accutom cutting or disc coring, even if large slices
up to 2mm were used. Therefore to be able to study the state of
the machined sub-surface by TEM analysis, a back thinning method
had to be used. The roughness of the intermediate machined
surface would not allow back thinning to electron transparency,
and thus the technique was applied to the flat, fine machined
surface. Back-thinning was carried out by sectioning the
machined bar in a different orientation to that used for the
cross-sections so that the machined surface would be on one face
of the eventual 3mm disc. Coring, followed by dimpling and ion
beam milling the back, non-machined face of the specimen was
carried out using the same techniques and conditions as detailed
in Sections 5.2.1.4 and 5.2.1.5, although only one ion beam was
used for milling and the two protecting brass rods were not
required. Due to lower residual stresses in the fine machined
surface only slight buckling of the thinned specimen occurred.

5.3 METHODS OF OBSERVATION
5.3.1 Macro View of Machined Surfaces
The topography of the machined surfaces was examined with a
Zetopan optical microscope set in the reflection optical mode,
and a Cambridge S250 scanning electron microscope using a 21kV
electron beam and secondary electrons to form the image.

5.3.2 Cross-section Examination of Machined Surfaces.
5.3.2.1 Median Cracks in the Coarse Machined Surface.
Prepared cross-sectional specimens were viewed with an MEF
optical microscope in the reflection mode at high magnification
using a Hg vapour lamp UV light source illumination and an
objective lens of 140 x magnification with numerical aperture of
1.3 (oil immersion n = 1.515). However, no features at the
machined surface were readily apparent. Using a non-text book method, cracks normal to the machined surface were made visible to a high degree of contrast and resolution with an oblique illumination. The technique involves closing down the field stop by a certain amount and using the shaded field of view where the subject is obliquely illuminated by the diffuse scattered light. This phenomenon was first seen by Rune Holm at the University of Surrey, when certain features appeared to stand out during an optical microscope examination of indentation cracks. Topographical features are highlighted by this "penumbra" illumination and remain in a bright field mode, but at a much decreased brightness. The effect is consistent whether Critical or Kohler illumination is applied. Median cracks were also examined with a scanning electron microscope using a 21 kV electron beam.

To study the mode of crack propagation backscattered electron analysis was used to highlight the ceramic microstructure and the crack path. To achieve a high magnification backscattered image while retaining good resolution, various conditions were tried with a Cambridge S 250 SEM. It was found that with a very high magnification and an acceleration voltage of 21 keV, the image resolution was poor due to the inefficient backscattered electron detectors which just collect backscattered electrons with no attracting voltage bias. If a compensating higher voltage was used, then the intergranular phase areas tended to flare and cause a loss of definition around the grain boundaries. Therefore a 21 kV electron beam and a magnification of 5,000 x to 10,000 x were used with working distance of 15mm and a frame record of 125s to reduce noise.

5.3.2.2 The Residual Compressive Layer in TEM Cross-Section.

Examination of the machined sub-surface layers was undertaken with a JEOL 200 CX transmission electron microscope using a 200kV beam. To measure the depth of the deformed compressive layer, TEM images were taken to reveal the extent of the dislocation damage in the microstructure. An alternative means of observation was also used whereby electron micro diffraction patterns of single grains revealed deformation in the crystalline structure via "streaking" or arcing of the diffraction spots. Arcing is a typical phenomenon that occurs in crystalline
materials that contain deformation (Edington 1976, Thomas and Goringe 1979). A similar effect can also be seen in Laue back reflection and transmission X-ray patterns of single crystal materials that contain deformation (Cullity 1967).

5.3.2.3 The Residual Compressive Layer, Back-Thinned TEM Specimen

Work by several authors has shown that the majority of dislocations in silicon nitride have Burgers vectors \( \mathbf{b} = \langle 0001 \rangle \). They are therefore seen in most tilts, except when \( \mathbf{g} \cdot \mathbf{b} = 0 \). Therefore they were searched for in a bright field imaging mode, and also by the invisibility criterion \( \mathbf{g} \cdot \mathbf{b} = 0 \), where \( \mathbf{g} \) is the operating reflection. Under this condition dislocations must go out of contrast for two non-parallel reflections. This would be done by selecting a large \( \beta - \text{Si}_3\text{N}_4 \) grain to obtain a good micro diffraction pattern. As the Burgers vectors are \( \mathbf{b} = \langle 0001 \rangle \) then most tilts would provide a correct reflection. A dim spot near the central transmitted spot is chosen, and the specimen is tilted until it reaches the same intensity as the central spot, thus giving a two beam condition. Looking at the bright field image for each case will highlight dislocations existent in the grain, and by indexing the pattern the Burgers vector is found. The process is repeated for three other spots in the pattern.

5.4 THE EFFECTS OF MECHANICALLY POLISHING THE MACHINED SURFACES AND A SURFACE CONTAINING AN INDENTATION

In removing a machined surface using a polishing process that introduces only a minimal amount of pull-out, evidence of localised damage underneath deep machining grooves can be seen in the form of remnant tracks (Section 6.3.1c and 6.3.2). By applying an indentation in the machined surface and measuring its depth, the layer by layer removal of a surface can be accurately monitored, thus allowing quantification of the depths to which the deformation extends. Polishing was carried out with a 6 micron diamond compound and oil based lubricant on a stationary tin lap.
5.5 EFFECT OF INDENTING A COARSE MACHINED SURFACE

Diamond machining leaves a surface with a long range biaxial residual stress field, with semi-elliptical/median cracks parallel to the machining direction under the compressive plastically deformed layer. The effect of the compressive and tensile stresses acting on the machining-induced cracks can be studied by applying a tensile stress to the machined surface, parallel and normal to the machining direction, in 3 point flexural rupture tests (Rice and Mechoslsky 1979, Marshall et al 1983). Another test that may reveal stress field/crack properties is the Vickers diamond indentation of a machined surface where the indentor is aligned orthogonal to the machining direction.

A section of the coarse machined surface was placed directly on the table of a Vickers macro hardness testing machine, without the use of cyanoacrylate adhesive for bonding. The specimen was aligned so that the machining direction would be parallel to the indentor diagonal edge. Via an internal lever system, the Vickers pyramid diamond indentor was automatically lowered on the specimen surface reaching peak load within 2s, with unloading taking place after about 6-8 seconds. Loads of 20kg, 50kg and 70kg were applied to the machined surface.

5.6 THREE POINT FLEXURAL RUPTURE TEST

To examine the effects of applying a tensile force to a machined surface a three point flexural rupture test (Figure 5.7a) was carried out at Rolls-Royce Leavesden Materials Laboratory. A longitudinally ground coarse machined bar was cleaned in an ultrasonic bath with Genklene and set up in a 10mm span three point flexural rupture test rig on a 1361 Instron Servo screw machine. The machine was fitted with an intelligent controller linked to a 10kN load cell, and this monitored the applied load during the test.

The bar was positioned on two 10mm span SiC rollers and aligned with two pins to set it perpendicular to the roller axes (Figure 5.7b) The rig was covered with a plastic bag to collect any fracture debris. A preload of 2.8% of 2 kN = 56 N was applied. The main load was then activated and applied linearly at 15kN/min, which is typical of the load appreciation rate
Figure 5.7a  Schematic diagram of 3 point flexural rupture test on a coarse ground longitudinally machined bar

Figure 5.7b  Schematic birds-eye view of test bar position on rollers
encountered by gas turbine blades during engine start up. Fracture occurred within seconds, and the load registered by the controller at the point of fracture was 44.3% of 2 kN = 886 N (90.3kg). The origin of fracture was viewed in cross-section with a SEM and an elemental examination was carried out with a JEOL 35 CF electron microscope using energy dispersive X-ray spot analysis and a 15kV electron beam.

5.7 SINGLE POINT SCRATCH TESTS

A longitudinally fine machined 3 point flexural test bar was bonded to a scratch machine table with cyanoacrylate adhesive, ensuring that the length of the bar would be parallel to the direction of motion of the indentor. The scratch machine consisted of a heavy steel base (Figure 5.8) upon which a microscope stage with a gramophone-type arm could be moved manually. A Vickers diamond pyramid indentor at the end of the arm was positioned so that the open face would be normal to the direction of scratching motion, and would therefore make contact with the material surface. This position would more closely resemble the conditions of a single diamond grinding wheel particle gouging the surface. Counter balance weights were adjusted so that the diamond tip would just touch the material surface. Weights were applied on top of the indentor and scratches were implemented at loads of 25, 50, 100, 200, 500, 750, 1000, 1500, 2000gm at a traverse speed of approximately 1cms⁻¹. The geometry of the machine with weights applied at the end of the counter balanced arm gave rise to a non-stiff scratch process.

Prior to observation with a SEM the scratched surface was not degreased and cleaned in acetone and methanol as this would have washed away the material debris. A thorough cleaning process was carried out before the scratches were implemented. To avoid charging occurring on the material fragments when under the electron beam in the SEM the scratched bar was given a long exposure in the gold coating unit. After the surface debris and grooves had been thoroughly examined and analysed, the bar was cut at various positions with an Accutom diamond cutting wheel normal to the scratch direction, to allow a comprehensive cross-sectional view of the grooves, sub-surface deformation and sub-surface cracks.
Figure 5.8 Schematic side-view diagram of scratching machine
CHAPTER 6. EXPERIMENTAL RESULTS

SUMMARY

This chapter details the results obtained in the study of machining damage present in coarse, medium and fine surface roughness finishes, together with interaction effects when these surfaces are mechanically polished. This is followed by single point scratch test results.

6.1 ANALYSIS OF COARSE MACHINED SURFACE

The surface profiles of longitudinally and transversely machined bars were measured and values of centre line average roughness of around 0.4μm were estimated. Macro features such as 2μm deep 18μm wide grooves present within general surface roughness were examined and compared to the effects the grinding conditions could theoretically have. A wedge cross-section view revealed some form of damage under a deep groove. Using the "penumbra illumination" optical microscope technique on normal cross-section specimens an examination clearly revealed sub-surface median cracks extending from 6 - 45μm below the machined surface. SEM and backscattered electron analysis produced images which were less highlighted with lower contrast, but which allowed the granular and intergranular crack propagation route to be viewed. All cracks in a number of specimens were then examined thereby allowing a statistical analysis of the length, distance from surface, frequency etc. TEM examination of the sub-surface revealed a large concentration of dislocations in the grain structure estimated to be around $4 \times 10^{14}$ m$^{-2}$. The innovative technique of examining the extent and position of distorted X-ray microdiffraction spots within the surface provided an approximate thickness measurement of the thin surface layer in residual compression. This was estimated to be 4 - 5μm deep. A parallel cross-section optical microscope examination, again with the penumbra technique, revealed median cracks to be semi-elliptical in shape extending from 19 - 101μm in length. Using the crack dimensions a prediction of bar fracture strengths is made.

6.2 ANALYSIS OF THE INTERMEDIATE MACHINED SURFACE

Surface profiles and centre line average roughness measurements
were again made. An unusual convex surface curvature of around 1m radius was found across the bar surface. This is evidence of the compressive biaxial field which is stronger normal to the machining direction. A SEM examination again revealed deep (1μm) surface grooves within the general surface roughness of 0.3μm.

6.3 ANALYSIS OF THE FINE MACHINED SURFACE

Profiles, surface roughness and a shallower convex surface curvature of 8m to 13m radius were measured. Optical and SEM examination revealed the presence of material pull-out "remnant tracks" of up to 16μm width and 1.5mm length on the shiny surface. These are evidence of machining damage left over from previous grinding stages. A cross-sectional optical and TEM examination revealed no cracks or distorted X-ray microdiffraction spots.

6.4 THE EFFECTS OF MECHANICALLY POLISHING THE MACHINED SURFACES AND A SURFACE CONTAINING AN INDENTATION

Polishing away the surface roughness of the coarse machined bars produced similar material pull-out remnant tracks as were found in the fine machined surfaces, and polishing an indented surface gave rise to a band of material pull-out around the indent site. This shows the interaction effects between material damage previously introduced and mechanical polishing. The number of small radial cracks around the indent were found to be greater beneath the surface than on top.

6.5 EFFECT OF INDENTING A COARSE MACHINED SURFACE

20 kg, 50 kg and 70 kg indentations were made on the surface of a coarse machined bar. The 70 kg indentation caused the bar to fracture and allowed a cross-section examination which revealed semi-circular plastically deformed zones, lateral cracks and median cracks. The fracture occurred parallel to the coarse machining direction and linked up the median/radial cracks of all three indents.
6.6 IMPURITY FRACTURE ORIGIN OF THREE POINT FLEXURAL RUPTURE TEST

A SEM examination of the fracture origin of a bar revealed an impurity defect 100µm long, 130µm below the bar surface. Energy dispersive X-ray microscopy showed the presence of Al and Cl impurities, which were not expected.

6.7 SINGLE POINT SCRATCH TESTS

The 3 point flexural rupture test bars were not cleaned after scratching in order to allow an analysis of the material debris on the surface. Surface and sub-surface deformation/fracture was found to increase in stages from the 25 gm though to the 2000 gm scratch load. It began as grain boundary fracture and a shallow groove, then fragments of material debris and scale-like cracks in the deeper groove bottoms. This was followed by larger compacted fragments, chevron cracks and finally chip fracture. A high pitched scratching noise was heard during the scratch tests.

The diamond indentor was found to be worn and contained highly compacted material debris which increased the tip roundness. Extensive manipulation of the penumbra lighting conditions and photographic processing proved successful in recording the nature of sub-surface median cracks in normal cross-section. They were found to initiate at the boundary of a semi-circular deformed zone situated under grooves, a similar situation to the initiation of cracks in the coarse machined sub-surface.
CHAPTER 6 EXPERIMENTAL RESULTS.

6.1 ANALYSIS OF THE COARSE MACHINED SURFACE.

6.1.1 Profile Analysis

Longitudinally Machined Bars

The surface profiles of five coarse longitudinally machined bars were recorded with a Talystep instrument. The bars were numbered 1 to 5 with 1 and 5 being measured in two different areas for measurements with traverse normal to the machining direction. The results are shown in Tables 6.1a. and 6.1b.
TABLE 6.1: SURFACE ROUGHNESS PARAMETERS FOR THE COARSE MACHINED SURFACE - LONGITUDINALLY MACHINED BARS

Table 6.1a: Traverse Normal to Machining Direction

<table>
<thead>
<tr>
<th>BAR</th>
<th>$R_a$ (μm)</th>
<th>$R_{max}$ (μm)</th>
<th>$R_{a_{max}}$ (μm)</th>
<th>$R_{a_{min}}$ (μm)</th>
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<tbody>
<tr>
<td>1</td>
<td>0.357 (1200)*</td>
<td>2.1</td>
<td>0.558 (200)</td>
<td>0.215 (200)</td>
</tr>
<tr>
<td>1</td>
<td>0.336 (800)</td>
<td>2.4</td>
<td>0.423 (200)</td>
<td>0.185 (200)</td>
</tr>
<tr>
<td>2</td>
<td>0.411 (1000)</td>
<td>2.2</td>
<td>0.610 (200)</td>
<td>0.265 (200)</td>
</tr>
<tr>
<td>3</td>
<td>0.312 (750)</td>
<td>1.8</td>
<td>0.343 (200)</td>
<td>0.265 (200)</td>
</tr>
<tr>
<td>4</td>
<td>0.352 (1075)</td>
<td>2.8</td>
<td>0.393 (200)</td>
<td>0.253 (200)</td>
</tr>
<tr>
<td>5</td>
<td>0.344 (1175)</td>
<td>2.4</td>
<td>0.373 (200)</td>
<td>0.268 (200)</td>
</tr>
<tr>
<td>5</td>
<td>0.360 (500)</td>
<td>2.3</td>
<td>0.455 (200)</td>
<td>0.273 (200)</td>
</tr>
</tbody>
</table>

OVERALL PARAMETERS FOR A COARSE MACHINED SURFACE - LONGITUDINALLY MACHINED

$R_a = 0.35(4)$ μm (6500)

$R_{max} = 2.8$ μm

$R_{a_{max}} = 0.61$ μm

$R_{a_{min}} = 0.19$ μm

Table continued on next page
### TABLE 6.1.b: Traverse Parallel to Machining Direction

<table>
<thead>
<tr>
<th>BAR</th>
<th>$R_a$ ($\mu$m)</th>
<th>$R_{\text{max}}$ ($\mu$m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.03</td>
<td>0.20</td>
</tr>
<tr>
<td>2</td>
<td>0.11</td>
<td>0.48</td>
</tr>
<tr>
<td>3</td>
<td>0.11</td>
<td>0.72</td>
</tr>
<tr>
<td>5</td>
<td>0.08</td>
<td>0.40</td>
</tr>
</tbody>
</table>

$R_a$ = Centre line average roughness

$R_{\text{max}}$ = Maximum peak to valley height, i.e. the largest single peak to valley height.

$R_{\text{ramax}}, R_{\text{ramin}}$ = Maximum and minimum CLA roughness in isolated regions; the sample lengths are in brackets.

* Note: All figures in brackets denote sample length used ($\mu$m).
The centre line average roughness of the coarse machined bars varied from ± 0.34μm to ± 0.41μm with an average of ± 0.35μm. A typical surface is shown in Figure (6.1a). The estimated value is close to the nominal figure of ± 0.4μm as given by P S Marsden Precision Engineering Ltd. Some isolated regions from 100μm to 300μm in length were found to have a rougher surface with a CLA number of up to ± 0.61μm. These regions were possibly due to enhanced material removal caused by inhomogeneities in the diamond wheel surface which apply localised excess contact pressure, or possibly inhomogeneities in the material itself, including the cellular network structure. There are other areas of around 100μm in length which have a CLA roughness down to ± 0.17μm. These regions are probably formed when the diamonds passing over that area are prevented from penetrating to the depth set in the depth of cut, by another region which is slightly higher, and just scrape the surface at a shallower depth. It is also possible that the diamonds on that particular circumference line on the wheel are small or temporarily worn and truncated.

Independent of the local surface roughness there exists a random distribution of a small number of deep machining grooves which have a maximum peak to valley height $R_{\text{max}}$ of 1.8μm to 2.7μm. Examining the profile in Figure 6.1a in detail, the $R_{\text{max}}$ value of the groove A is approximately 2.4μm. Magnifying the region with a vertical magnification at 20,000 x and a horizontal magnification of 2000 x the groove shape is clearly detailed (Figure 6.1b). The actual groove is approximately 2.0μm deep, 18μm wide and is surrounded by material pile-up on either side. A representative groove is shown in the reflection optical image, Figure 6.2.

The centre line average roughness with traverse parallel to the machining direction fell in a range from ± 0.03μm to ± 0.11μm, with an average of ± 0.08μm. A typical profile is shown in Figure 6.3. The maximum peak to valley heights vary from 0.20μm to 0.72μm, which is in the order of magnitude of the surface roughness measured normal to the machining direction.

Transversely Machined Bars.

The surface profiles from different areas of two transverse coarse machined bars were recorded using a Talystep instrument
Figure 6.1a Profile of coarse machined surface with traverse normal to the grinding direction

Figure 6.1b Magnified profile of deep groove A in the coarse machined surface
Figure 6.2  Reflection optical images of machined surface. A deep groove is visible in the centre. 0° tilt
Figure 6.3  Profile of coarse machined surface with traverse parallel to the grinding direction
with traverse normal to the machining direction. The profiles were analysed giving the estimated roughness parameters set out in Table 6.2, and a typical profile is shown in Figure 6.4a.

The CLA roughness for the surface varied from 0.38µm to 1.05µm with a mean of 0.69µm. These figures are approximately double those derived from the longitudinally coarse machined surface and are unexpected. Magnifying the profile in Figure 6.4a the large groove C can be studied in detail (Figure 6.4b). The groove is larger than those present in the longitudinally machined surface, and has a depth and width of approximately 2.6µm and 35µm respectively.

There are two possible explanations for the existence of such a rough surface: a) Coarse machining is carried out in two stages (See Coarse Machining Section 5.1.2.1). Initial grinding uses a 150 grit resin bonded diamond wheel, which is then followed by a 350 grit wheel, producing a surface of 0.35µm CLA roughness. Failure to employ the second stage may result in a surface of 0.69µm CLA roughness, b) As the hipped ceramic material is so hard, a maximum of 3 or 4 bars are machined at a time. Thus the 9 or 12mm of material that traverses under the diamond wheel is shorter than the 50mm for the longitudinally machined bars, and this may result in a change in the dynamics of the material removal process and perhaps a higher contact load.

While b) is a possible reason for the formation of a very rough surface, the increase in total force of the diamond wheel needed to cause all the diamonds to penetrate deeper should be quite substantial, and is unlikely to be provided by the disparity in material length. Therefore the former explanation is the more likely occurrence.
### Table 6.2: Surface Roughness Parameters for the Coarse Machined Surface—Transversely Machined Bars.

**Traverse Normal to Machining Direction**

<table>
<thead>
<tr>
<th>BAR</th>
<th>$R_a$ (µm)</th>
<th>$R_{max}$ (µm)</th>
<th>$R_a$ (µm)$_{max}$</th>
<th>$R_a$ (µm)$_{min}$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(200)</td>
<td></td>
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<td></td>
</tr>
<tr>
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<td>0.694 (1000)</td>
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<td>0.505</td>
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<tr>
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<td>0.375</td>
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<td>1.045</td>
<td>0.430</td>
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<td></td>
<td>0.723 (1500)</td>
<td>2.4</td>
<td>0.980</td>
<td>0.390</td>
</tr>
<tr>
<td>Parameters for whole Bar</td>
<td>0.720 (3000)</td>
<td>3.2</td>
<td>1.045</td>
<td>0.390</td>
</tr>
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</table>

**Overall Parameters for a Coarse Machined Surface—Transversely Machined Bars**

- $R_a = 0.69$ (µm) (7000)
- $R_{max} = 3.20$ (µm)
- $R_{a_{max}} = 1.05$ (µm)
- $R_{a_{min}} = 0.38$ (µm)

Note: Figures in brackets denote sample length (µm).
Figure 6.4a

Figure 6.4b  Magnified profile showing groove C in detail

Figure 6.4  Profile analysis of coarse machined surface, transversely machined bar
6.1.2 SEM EXAMINATION

In Figure 6.5 it can be seen that pile-up has been caused by the action of plastic flow, where the material has been pushed out by a singularly large diamond and has been superposed on the surrounding lower level of surface damage, leaving a large groove with a compacted bottom. Viewing at a shallow angle the shape of the groove is seen to be non-symmetrical (Figure 6.6). It is therefore inevitable that the underlying sub-surface deformation zone, which is deeper than the general level of sub-surface damage, and the median crack area are also un-symmetrical. Consequently the residual stress components associated with the damage site will be distorted. The "depression" B in Figure 6.1a is 1µm deep, 90µm wide and will probably contain a less intense and shallower residual field than groove A, due to the shallow depth and greater width of the underlying deformation zone. Therefore the layer of residual compression formed by the overlap of neighbouring damage sites (coupled with the localized compressive stress acting at the zone boundary), have a greater closing effect on the residual tensile crack-opening force than for A (although the localized compressive stress in A is greater). Furthermore, it is likely that only single narrow grooves created by a wedge opening force contain a localized concentrated stress, sufficient to nucleate a median crack parallel to the machining direction.

In Figure 6.7 the effect of the competing process between the compressive and tensile fields is seen in the formation of a delamination crack which exists to depths of 3.0 - 12µm.

6.1.3 Wedge Cross-Section View.

A small section of a longitudinally machined bar was cut with an Accutom cutting machine, using a 350 grit resin bonded diamond cutting wheel. The machined surface was lapped with a 3mm wide flat lapping wheel to a depth of 10µm, on a Dimpler machine, and this produced a curved slope outside the 3mm diameter central flat area (Figure 6.8a). The slope provided a wedge cross-section view of the machined surface and allowed an analysis of the surface and sub-surface layers. A large groove approximately 2µm deep, was found to contain a line of sub-surface damage in the form of grain pull-out (Figure 6.8b). The damage extends from just beneath the groove (Figure 6.8c) to the
Figure 6.5  Pile-up around the machining groove is caused by the volume being accommodated by the surrounding material via the action of plastic flow. 0° tilt
Figure 6.6 Unsymmetrical shape of deep machining groove. 75° tilt
Figure 6.7  Cross-section view of machined surface showing sub-surface delamination at depths of 2.5 - 18μm. 5.7° tilt
Figure 6.8a  Reflection optical image of flattened area relative to the original machined surface.

Figure 6.8b  Groove with sub-surface damage in the form of grain pull-out.

Figure 6.8c  Edge of machined surface showing groove tip.
flat central area 10µm deeper, and is consistent with the position in which median sub-surface cracks are expected to exist. The line of sub-surface damage was traced to a depth of 10µm and along, parallel to the groove, it was measured to be about 230µm in length; however, these may both extend further.

6.1.4 Normal Cross-Section Examination of Coarse Machined Surface

6.1.4.1 Optical Microscope Examination

Examining the sandwich cross-section interface with penumbra illumination revealed the presence of sub-surface median cracks. The median crack in Figure 6.9 is resolved with good contrast and appears to initiate at a depth of approximately 5.0µm from the free surface (A key to the features is given in the schematic in Figure 6.10). One side of the crack is dark which confirms that the image is given by an oblique illumination of the surface. It is important to note that the crack does not extend from underneath a machined groove, but from a flat surface. The crack probably did initiate from a groove but then a thin layer of material may have been removed on the next passing of the wheel, leaving some sub-surface deformation and the crack. From the free surface to the start of the crack there appears to be a tributary system of microcracks. Expanding the area to a total magnification of 5699 x (causing some empty magnification), and focusing into the specimen to a depth of 0.5µm, the median crack becomes slightly defocussed but the microcracks are clearly resolved (Figure 6.11). This slight curvature at the ceramic/epoxy interface is due to preferential polishing of the epoxy adhesive during the specimen preparation stage. The tributary system is estimated to be approximately 15-16µm wide and this is consistent with a typical groove width. Measuring with graduated optical eyepieces the crack depth from the free surface to the tip of the crack was estimated to be 23.7µm.

Some damage sites contain multi cracks where a median crack is usually accompanied by smaller cracks at larger angles to the normal. The images in Figure 6.12 are less clear as some difficulty was found in achieving the correct illumination conditions. Nevertheless, it can be seen that the damage characteristics more closely resemble a single point scratch
Figure 6.9 Reflection optical image of interface using penumbra illumination. A sub-surface crack lies roughly normal to the machined surface. 0° tilt
**Figure 6.10** Schematic of optical image in Figure 6.9, with key to relevant features

A = Point of initiation of crack
B = Tributary system of microcracks
\[\cdots\cdots\] = Machined surface
Figure 6.11 Tributary system of microcracks that converge from crack initiation. 0° tilt
Figure 6.12a

Figure 6.12b

Figure 6.12  Reflection optical images of multi crack damage sites with corresponding (key) schematic diagrams (A = cracks, B = deformed zone, C = bright field illumination region).
damage site (See Section 6.7.3) where the deformed zone is semi-circular with cracks initiating along the plastic/elastic boundary.

6.1.4.2 SEM Examination

Examination of the sandwich cross-section interface with scanning electron microscopy shows the width of the epoxy interface and the slight curvature due to the preferential polishing of the adhesive (Figure 6.13). Median sub-surface cracks normal to the free surface were not readily visible in a random search. To locate them they were first found with the optical microscope under the special penumbra illumination conditions, and the positions along the interface were measured. The appearance of the cracks is in contrast to the well defined images under the optical illumination. The crack shown in Figure 6.14 is slightly open only in a few places along the length, and is actually seen due to the burning effect of the hydrocarbons that remained in the surface after the acetone treatment in the specimen preparation stages. As far as can be resolved the initiation point of the crack is measured to be about 5.5μm to 8.5μm from the free surface (Figure 6.15). In comparison with cracks formed by Vickers hardness indentations the crack can be deemed as nearly closed. This suggests that the crack may have been closed as a result of specimen preparation surface polishing, which left micro stresses in the surface (Mecholsky et al 1977), similar in nature to the machining stresses being investigated. It is therefore feasible that the cracks are visible under optical illumination due to the penetration of light, which illuminates the sub-surface regions (the optical transparency of silicon nitride was also exploited in the observation of macro defects existent in the matrix structure (See Section 3.2.1.1 and 3.3.1.1) This was confirmed when the cracks were no longer visible under the optical penumbra illumination after the specimen surface had been coated by gold. The highly reflecting surface does not allow the penetration of light.

A direct comparison of the two imaging methods is shown in Figure 6.16. The secondary electron image was recorded at zero degrees tilt, which at high magnification causes an atomic number
Figure 6.13  Secondary electron image of epoxy interface.  45° tilt
Figure 6.14 Mosaic construction of sub-surface crack. Only parts of the crack are open at the specimen surface. 45° tilt
Crack initiation region near the machined surface. 45° tilt.
Figure 6.16a. Reflection optical image of sub-surface crack. 0° tilt.

Figure 6.16b. Secondary electron image of same crack showing diminished resolution at crack tip. 0° tilt.
contrast effect to be superimposed with topographical contrast. Although there is an improvement in resolution compared to the images taken at 45° tilt, the crack is still diffuse.

6.1.4.3 Backscattered Electron Analysis of Crack Path

The sub-surface median cracks were analysed by back scattered electron imaging and an improvement in resolution and contrast was achieved as compared to the secondary electron images. However, a direct comparison of the crack images formed by penumbra illumination and back scattered electron collection shows that the crack is still highlighted to a greater extent with the low intensity oblique optical illumination (Figure 6.17). Nevertheless, back scattered electron analysis can provide a valuable means to study the mode of crack propagation.

As far as can be resolved the crack in Figure 6.18 taken under the optimum imaging conditions, appears to follow an intergranular path. Other cracks also displayed an intergranular route, although in a few cases some transgranular fracture did appear to exist. The crack shown in Figure 6.16b may be an example where at the point c it cleaves a β-grain (Figure 6.19).

6.1.4.4 Statistical Analysis of Sub-Surface Cracks

A statistical analysis of sub-surface cracks was carried out on three sandwich cross-section specimens taken from the same machined bar, two 450μm thick and one dimpled to a central thickness of 20μm. Each interface surface was analysed, therefore a total of six positions along one machined bar were sampled. It was immediately noted that many cracks did not extend exactly normal from the machined surface, but were either inclined at an angle or were curved. Therefore in crack depth measurements, the distance was taken from the free surface directly normal to the crack tip, not along the crack which would give the crack length (Figure 6.20). Also it appears that there may be a directionality in the crack inclination, where under one machined surface 50% were inclined to the left and only 21% to the right. The calculation was made by considering any crackinclined more than 5° from the normal as being a "deviated" crack. The left-right classification of the crack inclination is of course arbitrary and will vary for each cross-section specimen as the surface to be prepared for analysis was randomly
**Figure 6.17a** Reflection optical micrograph of sub-surface crack. 0° tilt

**Figure 6.17b** Backscattered electron image of same crack. 0° tilt
Figure 6.18  Backscattered electron image mosaic construction of sub-surface crack taken at a magnification of 10,000 x. 0° tilt
Figure 6.19  Secondary electron image of crack taken at a magnification of 10000 x.  0° tilt
Figure 6.20 Schematic diagram of cross-sectional view of machined surface showing curved sub-surface crack and corresponding dimensions. Not drawn to scale.
chosen. However, the relation between two machined surfaces joined together in one cross-section specimen is very important as it may give an indication of any extraneous effects. Some crack sites contained more than one crack, and each one was measured and entered as a separate crack in the results. The crack parameters are given in Table 6.3. It is noted that the crack dimensions for the dimpled specimen are larger than the two 450μm thick ones. Both the dimpled specimen interface surfaces have much greater maximum crack depths of 45.7μm, and also greater average crack depths of 18.3μm and 15.8μm. This may lead one to believe that the dimpled specimen suffered mechanical damage during preparation which caused the original cracks to propagate deeper. It is also possible that this specimen slightly buckled due to the transverse residual compressive stresses of the order of -200 MPa, coupled with the longitudinal compressive stresses of up to -155 MPa (measurements of stresses using X-ray diffraction by P E J Flewitt (1989)). However, it cannot be discounted that as thin slices of silicon nitride are transparent to light, the cracks may have been resolved to a greater definition.

The crack initiation depth for the six machined interface surfaces ranges from 1.6-8.7μm with an average of 4.4μm (±σ = 0.8μm), and therefore the average depth of the compressive layer can be taken as approximately 4-5μm. Furthermore from a polished 7° oblique section of the machined surface, Quinn (1989) found a layer of mechanical deformation ranging from 2.4 - 9.8μm with an average of 4.0μm. The depth of 10μm which was derived by line force and X-ray measurements on bars ground with a 240 grit diamond wheel (Johnson-Walls et al 1986) is reasonably consistent with this.

Some evidence of possible directionality in the crack inclination can be seen in the dimpled specimen numbers. At interface A 47% of the cracks are inclined to the left whereas only 18% are inclined to the right. The two bar sections which formed the original sandwich were positioned with the grinding directions in opposition.* Therefore, if the interface A had a directionality towards the left, then the interface B should also contain a directionality towards the left. This appears to be the case where 50% of the cracks are inclined to the left and 21% * See Figures 5.7a, 5.5a.
### TABLE 6.3: CRACK PARAMETERS

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<thead>
<tr>
<th>SPECIMEN</th>
<th>INTERFACE</th>
<th>NUMBER OF CRACK SITES WITH MULTI CRACKS</th>
<th>NUMBER OF CRACK SITES</th>
<th>TOTAL NUMBER OF CRACKS</th>
<th>LARGEST CRACK DEPTH (µm)</th>
<th>SMALLEST CRACK DEPTH (µm)</th>
<th>AVERAGE CRACK DEPTH (µm)</th>
<th>LARGEST CRACK INITIATION DEPTH (µm)</th>
<th>SMALLEST CRACK INITIATION DEPTH (µm)</th>
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<td></td>
<td>B</td>
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<td>8</td>
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<td>12.0</td>
<td>5.5</td>
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</table>

| SPECIMEN | INTERFACE | AVERAGE CRACK SITE SEPARATION (µm) | PERCENTAGE OF CRACKS INCLINED TO MAXIMUM ANGLE OF AVERAGE ANGLE OF INCLINATION (DEG) |
|----------|-----------|-----------------------------------|-----------------------------------------------------------------|---------------------------------------------------------------|
|          |           | LEFT                              | RIGHT                            | NORMAL                        | LEFT                        | RIGHT                      | LEFT                      | RIGHT                        |                |           |
| 1        | A         | 267                               | 38                               | 38                            | 25                          | 13.3                       | 13.1                      | 10.2                        | 8.1                        | 9.6       |
|          | B         | 267                               | 50                               | 38                            | 13                          | 25.7                       | 11.7                      | 12.6                        | 9.2       |
| 2        | A         | 183                               | 33                               | 50                            | 17                          | 24.0                       | 14.6                      | 11.8                        | 9.2                        | 4.9       |
|          | B         | 306                               | 33                               | 17                            | 50                          | 20.8                       | 5.0                       | 14.3                        | 4.9       |
| DIMPLED  | A         | 168                               | 47                               | 18                            | 35                          | 25.5                       | 23.6                      | 12.1                        | 9.0                        | 14.2     |
|          | B         | 168                               | 50                               | 21                            | 29                          | 50.4                       | 27.3                      | 9.0                         | 14.2     |
to the right at interface B. This observation however, is in contradiction with the results from the other interfaces. It is most likely that crack directionality is random and depends on the elastic/plastic contact-diamond shape, rather than an instability in the machining process (See unsymmetrical groove Section 6.1.2).

6.1.4.5 TEM Examination

Dimpled specimens were argon beam milled to electron transparency as described in Section 5.2.1.5. However, even with the protective shielding rods the epoxy adhesive still tended to be preferentially thinned and was not present in the centre of the specimens. Severe buckling also occurred in the centre, and this has been observed before where even a 0.25µm diamond polish caused severe buckling in alumina (Mecholsky et al 1977). Therefore just the areas further to the sides of the interface where buckling did not occur were used. Further milling was carried out to spread the central hole to these areas and achieve electron transparency. The hole later proved to be a valuable aid in allowing measurement of the compressive layer depth.

Damage at the machined surface was present in such concentration that individual dislocations in the β-grains were not resolvable in most areas (Figure 6.21). Nevertheless the thickness of the deformed layer may have been indicated by tracing the depth to which the dark dislocation concentration extended from the free surface. In practice this proved to be very difficult as the electron transparency reached such a low level at a distance of 5µm - 10µm from the free surface that the desired observation of dislocation free grains was not obtainable. Furthermore, the microscope did not have a measuring capability on the viewing screen and eye view calculations would have been necessary. Therefore an alternative means of measuring material deformation was required, and this was carried out by using X-ray microdiffraction analysis of the crystalline β-grains. Measurements of distance from the free surface were made possible by using the hole caused by the Argon beam milling.

The high mechanical deformation and large lattice strains in the crystalline grains at the machined surface resulted in severe distortion of the diffraction spots (Figure 6.22a).
Figure 6.21 TEM image of coarse machined surface showing a high concentration of damage
Figure 6.22a  X-ray microdiffraction pattern at coarse machined surface, with streaking of the diffraction spots

Figure 6.22b  At a distance of 4.0 - 4.5μm from the machined surface the arcing of the spots rapidly diminishes
phenomenon of "streaking" in diffraction spots is common in crystalline materials where an excess of edge dislocations can be said to cause lattice bending. A series of microdiffraction patterns were taken along the machined surface, each with its corresponding selected area image. By continuing to take patterns along the edge of the ion beam milled hole moving deeper into the machined surface the angle of distortion and therefore the extent of lattice deformation was found to decrease (Figure 6.22b). Each corresponding selected area image position was then mapped on an overall image of the examined region, therefore allowing measurements of distance from the free surface. The angle of distortion was found to rapidly decrease at a depth of 4.0μm and disappear at approximately 4.5μm. Thus the layer of residual compression is estimated to have an average depth of 4.5μm.

X-ray microdiffraction analysis can also be a useful tool in making a rough estimate of the dislocation density in individual β-grains. Assuming that an excess of edge dislocations of one sign cause lattice bending in the grains then the dislocation density \( B = \frac{1}{R b} \) where \( b = \) Burgers vector and \( R = \) radius of curvature of the bent grain (See below).

\[
B = \frac{1}{R b}
\]

As \( a = R \theta \) then \( B = \frac{\theta}{a b} \).

\( a = \) grain size

\( R = \) radius of curvature

\( \theta = \) included angle at the centre of curvature.

A calculation of the Burgers vector was not possible as the severe distortion of the spots would not allow indexation. However work by various authors (Butler 1971, Evans and Sharp 1971 and 1972, Kossowsky 1973), on different types of silicon nitride showed that the majority of dislocations have a \( \langle 0001 \rangle \) type Burgers vector, although in some cases an \( a + c \) axis
component is present. Therefore the magnitude of the \( q \) \( \beta \) vector in a \( \beta \)-grain can be taken as 2.911 Å. X-ray microdiffraction patterns were only taken from the largest grains to avoid sampling several grains together and causing an overlapped pattern. Therefore in a grain typically 3 - 4μm in length (of aspect ratio 3 or 4:1), where the included arcing angle \( \approx 24^\circ \), the dislocation density

\[
B = \frac{0.42}{2.911 \times 10^{-10}, 3.5 \times 10^{-6}} \approx 4.1 \times 10^{14} \text{ m}^{-2}.
\]

This is reasonably consistent within an order of magnitude to previous calculations on deformed \( \beta - Si_3N_4 \) crystals (Lee and Hilmas 1989).

6.1.5 Parallel Cross-Section Examination of Coarse Machined Surface

6.1.5.1 Optical Examination

Examination of cross-sections of the coarse machined surface cut parallel to the grinding direction revealed the presence of semi-elliptical sub-surface cracks. Some were visible under normal optical illumination (Figure 6.23a), while others required a low intensity oblique penumbra illumination (Figure 6.23b). Their shape and position is consistent with machining damage that runs parallel and normal to the grinding direction (Rice and Mecholsky 1979, Marshall et al 1983). The crack in Figure 6.23b is typical of a number of semi-elliptical cracks that are not completely symmetrical. This is due to the fact that loading during grinding creates a combination of axisymmetric indentation damage and linear deformation-fracture, and the distortion of cracks in this plane signifies a degree of directionality in the machining process.

The crack dimensions were measured and are as follows: the crack length varied from 100.8μm to 18.9μm with an average of 42.0μm, and the crack depth varied from 11.0μm to 3.2μm with an average of 7.3μm. However, the crack dimensions cannot be used for the prediction of fracture strengths as the depths have too great a statistical variation. The curvature of median cracks as seen in normal cross-section in Section 6.1.4.1 and 6.1.4.4, leads to the crack being exposed at different heights when cut and viewed in parallel (Figure 6.24). Nonetheless, it was possible to
Figure 6.23a Elongated semi-elliptical sub-surface crack, 101μm long, 11.0μm deep, under normal illumination

Figure 6.23b Semi-elliptical sub-surface crack, 23.6μm long, 7.9μm deep, under Penumbra illumination

Figure 6.23 Parallel sandwich cross-section of coarse machined surface
Figure 6.24  Schematic normal cross-section view of median sub-surface crack. When sectioning parallel to the machining direction the cut may occur at any position along the curvature. Thus a cut at B would expose a deeper crack than one at A.
measure the frequency of machining cracks along the grinding direction, and the average crack separation was found to be around 413μm. The average crack site separation measured from the normal cross-sections of the machined surface was found to be around 227μm. The area concentration of machining cracks is therefore estimated to be around 18/cm².

6.1.6 Prediction of Fracture Strength

A prediction of the order of magnitude of fracture strength of the transverse coarse machined surface can be estimated by using the size of the median cracks measured in Chapter 6.1.4.4. These cracks undergo a tensile stress when a transverse ground bar undergoes a three point bend test. As the stress intensity factor of machining induced cracks is very complicated and as yet unsolved, the $K$ value for an edge crack in a finite width sheet subjected to three point bending will be used (Rooke and Cartwright 1976). Thus the effect of residual stresses acting on the crack will not be taken into consideration.

The stress intensity factor $K_I$ is given by:

$$K_I = A \sigma c^{1/2}$$

where $A = 1.1$, $\sigma$ is the fracture strength and $c$ is the crack depth. Assuming simply that there is no precursor stable crack extension before an unstable configuration is attained the critical condition is given by:

$$K_I = K_c$$

$$A \sigma c^{1/2} = K_{IC}$$

where $c_o$ is the initial crack depth.

Thus $\sigma = \frac{K_{IC}}{Ac_o^{1/2}}$

The critical stress intensity factor $K_c$ was estimated by indentation fracture measurements carried out at the University of Surrey by R. Holm (Private communication) and was found to be 4MPa m$^{1/2}$. Using the deepest measured crack from the six positions taken along the coarse machined three point test bar ($c_o =$
45.7 \mu m, the fracture strength \( \sigma \approx 540 \) MPa. The transverse fracture strengths measured by Mr R Quinn at Rolls-Royce Leavesden ranged from 402-592 MPa with a modal value of about 525 MPa (Quinn 1979). The figure is at the upper end of the range and this may be due to the tensile residual component on the sub-surface median crack not being taken into account. Marshall et al (1983) measured the extent of stable crack growth during failure for a series of machined and indented HPSN surfaces. Using the ratio \( \frac{C_m}{C_0} = 5 \), where \( C_m \) is the crack depth at the point of failure, the fracture strength \( \sigma = 240 \) MPa. This figure is rather low, which could signify that either there is very little crack extension of the deepest cracks before failure, or there is none at all.

The fracture strength derived from the largest measured crack should be compared to the modal value of fracture strength data, as the cracks were only taken from one test bar. Other bars may have larger or smaller cracks, thus by statistics, on average, the largest crack from the single test bar will correspond to the most probable size of crack for many test bars. The mean strength value was found to be 508 MPa and an estimated fracture strength of 540 MPa based on measured crack depths shows good consistency.

6.2 ANALYSIS OF THE INTERMEDIATE MACHINED SURFACE

6.2.1 Profile Analysis

The surface profiles of two supplied longitudinally and transversely machined bars were each recorded in 3 areas using a Talystep instrument, with traverse normal to the machining direction. The profiles were analysed giving the roughness parameters in Tables 6.4a and 6.4b.
TABLE 6.4a: SURFACE ROUGHNESS PARAMETERS OF INTERMEDIATE MACHINED SURFACE - LONGITUDINALLY MACHINED BAR

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<thead>
<tr>
<th>$R_a$ (µm)</th>
<th>$R_{max}$ (µm)</th>
<th>$R_{a\ max}$ (µm)</th>
<th>$R_{a\ min}$ (µm)</th>
<th>Radius of Curvature (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.314(1550)</td>
<td>1.40</td>
<td>0.455</td>
<td>0.230</td>
<td>0.9</td>
</tr>
<tr>
<td>0.294(1200)</td>
<td>1.15</td>
<td>0.385</td>
<td>0.210</td>
<td>1.2</td>
</tr>
<tr>
<td>0.297(1350)</td>
<td>0.95</td>
<td>0.335</td>
<td>0.263</td>
<td>1.2</td>
</tr>
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</table>

OVERALL PARAMETERS FOR LONGITUDINALLY MACHINED BAR

- $R_a = 0.303 \text{ µm (4100)}$
- $R_{max} = 1.40 \text{ µm}$
- $R_{a\ max} = 0.455 \text{ µm}$
- $R_{a\ min} = 0.210 \text{ µm}$

TABLE 6.4b: SURFACE ROUGHNESS PARAMETERS OF INTERMEDIATE MACHINED SURFACE - TRANSVERSELY MACHINED BAR

<table>
<thead>
<tr>
<th>$R_a$ (µm)</th>
<th>$R_{max}$ (µm)</th>
<th>$R_{a\ max}$ (µm)</th>
<th>$R_{a\ min}$ (µm)</th>
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<tbody>
<tr>
<td>0.276(1500)</td>
<td>1.10</td>
<td>0.408</td>
<td>0.175</td>
</tr>
<tr>
<td>0.263(1500)</td>
<td>1.00</td>
<td>0.330</td>
<td>0.225</td>
</tr>
<tr>
<td>0.252(1500)</td>
<td>1.00</td>
<td>0.310</td>
<td>0.195</td>
</tr>
</tbody>
</table>

OVERALL PARAMETERS FOR TRANSVERSELY MACHINED BAR

- $R_a = 0.264 \text{ µm (4500)}$
- $R_{max} = 1.10 \text{ µm}$
- $R_{a\ max} = 0.408 \text{ µm}$
- $R_{a\ min} = 0.175 \text{ µm}$

Note: Figures in brackets denote sample length (µm).
A low magnification profile of the longitudinally machined surface (Figure 6.25) reveals a convex surface curvature. It can be proposed that this has been formed as a result of the strong compressive stress in the machined surface which acts normal to the machining direction. Its presence can be understood by considering the edge dislocation model proposed by Badrick et al (1979) where the machined surface can be considered to contain a dipole array of edge dislocations causing an expansion of the surface. This curvature was not detected in the coarse machined surface as the CLA roughness is of the same magnitude as the curvature height. Assuming simply that the profiled surface approximates to an arc of a regular circle, a rough estimate could be made of the radius of curvature, r, using the formula:

\[ r = \sqrt{\frac{(\frac{x}{2})^2 + y^2}{2y}} \]

where x is the width of arc and y the height.

Three measurements were taken on the bar and the estimated radius of curvature from 2mm sample lengths was found to be 0.9m, 1.2m, 1.2m, where the arc height across the 2.3mm surface width was approximately 0.8μm.

The CLA roughness of the longitudinally and transversely machined surfaces are 0.30μm and 0.26μm respectively, higher than the nominal figure of 0.1/0.2μm as given by P S Marsden Precision Engineering Ltd (see Section 5.1.3). Although there is a small difference in the roughness levels, a consistent difference between the transversely and longitudinally machined surfaces could not be ascertained due to a lack of specimens.

Machining grooves deeper than the general level of surface roughness are still present in this regime of surface finish (Figure 6.26a). Therefore they probably continue to play a dominant role in the strength of the ceramic bars, as for the coarse machined surface. Magnifying the profile in Figure 6.26a, the two grooves A and B are seen to retain the consistent shape of deep machining grooves, which are surrounded by material pile-up (Figure 6.26b). Groove A is approximately 1.1μm deep, and B is 0.7μm deep.
Figure 6.25 Surface profile of intermediate machined surface, longitudinally machined bar, revealing a convex surface curvature
Figure 6.26a Magnified profile showing grooves A and B in detail

Figure 6.26b Magnified profile showing grooves A and B in detail

Figure 6.26 Profile analysis of intermediate machined surface, longitudinally machined bar
6.2.2 SEM Examination

Typical grooves shown in Figures 6.27, 6.28 reveal that the groove shape is not only shallow, but also flat-bottomed, compared to the pointed groove left in the coarse machined surface. This suggests that the smaller 400 grit diamond grinding particles are more truncated in shape, and this is borne out by the symmetry of the deep grooves. Due to this and a smaller diamond particle size the extent of material pile-up is less than in the coarse machined surface (the volume of material plastic flow displacement is smaller), and the resultant deep machining grooves widths range from 3.5 to 4.0μm. There appears to be a roughly proportional increase in groove width with increasing maximum diamond grit size: the intermediate machined surface machined with a 15/08μm particle size (600 grit wheel [Table 5.1]) produced deep machining grooves of approximately 3.5 - 4.0μm widths, and the coarse machined surface machined with a 40/20μm diamond particle size (350 grit wheel), about 2.7 x higher, produced deep machining grooves of approximately 15μm width, 4.0 x higher.

6.3 ANALYSIS OF THE FINE MACHINED SURFACE

6.3.1 Profile Analysis

A) Cup Wheel Machined - Fine Finish

A profile analysis of three bars was carried out normal and parallel to the proposed machining direction. Centre line average roughness parameters were estimated and are entered in Table 6.5.
Figure 6.27a Material pile-up is lower than for the coarse machined surface. 0° tilt

Figure 6.27b The groove is shallower than the grooves in the coarse machined surface, and has a flattened bottom. 75° tilt

Figure 6.27 A typical machining groove on the intermediate machined surface
Figure 6.28a The distance between the grooves is random.  0° tilt

Figure 6.28b There is less pile-up around the groove which is shallower than the coarse machined surface grooves.  75° tilt

Figure 6.28 Typical groove in intermediate machined surface with flattened bottom as compared to pointed grooves existent in the coarse machined surface
TABLE 6.5: SURFACE ROUGHNESS PARAMETERS FOR THE FINE MACHINED SURFACE - CUP WHEEL MACHINED

**Traverse Normal to Machining Direction**

<table>
<thead>
<tr>
<th>BAR</th>
<th>$R_a$ (µm)</th>
<th>$R_{max}$ (µm)</th>
<th>$R_a_{max}$ (µm)</th>
<th>$R_a_{min}$ (µm)</th>
<th>Radius of Curvature (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0025</td>
<td>0.023</td>
<td>0.006(30)</td>
<td>0.002(30)</td>
<td>3.1</td>
</tr>
<tr>
<td>2</td>
<td>0.0038</td>
<td>0.058</td>
<td></td>
<td></td>
<td>1.6</td>
</tr>
<tr>
<td>3</td>
<td>0.0130</td>
<td>0.122</td>
<td></td>
<td></td>
<td>1.7</td>
</tr>
</tbody>
</table>

**Traverse Parallel to Machining Direction**

<table>
<thead>
<tr>
<th>BAR</th>
<th>$R_a$ (µm)</th>
<th>$R_{max}$ (µm)</th>
<th>$R_a_{max}$ (µm)</th>
<th>$R_a_{min}$ (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0045</td>
<td>0.026</td>
<td>0.006</td>
<td>0.003(150)</td>
</tr>
<tr>
<td>3</td>
<td>0.0038</td>
<td>0.055</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Note: Figures in brackets denote sample length (µm).

Figure 6.29a is a typical profile of the fine machined surfaces with traverse across the width of the bar. Roughness is greatest at the centre (Figure 6.29b) and similar to the intermediate longitudinally machined bars, the surface contains a convex curvature. The centre surface roughness may be due to the action of the cup grinding wheel which was not scheduled to be used, as discussed in Section 5.1.2.2. As for the intermediate machined surface, using the simple assumption that the curved surface approximates to an arc of a circle, a rough estimate was
Figure 6.29a Profile of fine machined surface with traverse across the width of the bar, normal to the proposed machining direction

Figure 6.29b Magnified profile of centre showing greater surface roughness
made of the radius of curvature. Using measurements from 2mm sample lengths, the radius of curvature for the three bars was found to be 3.1, 1.6 and 1.7m, with arc heights of approximately 0.4μm across the 2.3mm surface width.

The average centre line average roughness for the three bars was estimated to be 0.005μm, which is 1/10th of the nominal figure given by P S Marsden Precision Engineering Ltd. The data does not show any significant difference between traverse normal or parallel to the proposed machining direction, and this is probably due to the polishing action of the cup grinding wheel.

B) Cup Wheel Machined - Rough Finish

The surface profiles of four 12mm long pieces were recorded with traverse across the 3mm width of the bar, normal to the proposed machining direction. These surfaces had also been incorrectly machined and in some areas a diagonal lay is apparent (Figure 6.30a), giving a typical profile as shown in (Figure 6.30b). The CLA roughness of these areas measured from the four sections was estimated to be 0.035 microns. It is noted that these surfaces have a concave and not a convex curvature, the latter being a common characteristic of longitudinally machined surfaces. The radius of curvature for two areas was estimated to be approximately 2.4m and 2.5m, with arc depths of approximately 0.24μm across the 2.3mm surface width. Polished areas with no detectable lay also have a concave surface curvature and two areas gave higher figures of 8.1m and 13.4m.

The existence of patches of rough areas with a defined diagonal probably derives from a cup wheel fine machining process that did not polish the original surface uniformly.

C) Longitudinally Machined

The surface profiles of two correctly machined 15mm sections were recorded with traverse across the width of the bar, normal to the machining direction (Figure 6.31a,6.31b). The typical characteristic of a convex surface curvature in longitudinally machined bars is present in both surfaces and the radius of curvature was estimated to be 9.0 and 12.6 microns, with arc heights of approximately 0.065μm across the 2.3mm surface width of the bar. To estimate the CLA roughness the bars were each
Figure 6.30a The surface has been left with a "rough" finish and the machining has a diagonal lay. $0^\circ$ tilt

Figure 6.30b Surface profile of bar with traverse across width of bar, revealing a concave surface curvature

Figure 6.30 Analysis of the fine machined surface, machined with a cup wheel
Figure 6.31a Low magnification surface profile showing a convex surface curvature and evidence of "grooves" or "tracks"

Figure 6.31b Low magnification surface profile of a different bar. The position of the machining "tracks" is random.

Figure 6.31c Magnified profile of surface in Figure 6.31b showing "track" in detail.

Figure 6.31 Profile analysis of fine machined surface, longitudinally machined bars.
analysed at a higher magnification in three sections, allowing them to be tilted accordingly to produce horizontal profiles. The derived roughness parameters are presented in Table 6.6.

<table>
<thead>
<tr>
<th>Grooves Included in Analysis</th>
<th>Grooves Not in Analysis</th>
<th>Radius of Curvature</th>
</tr>
</thead>
<tbody>
<tr>
<td>Section</td>
<td>$R_a$</td>
<td>$R_{max}$</td>
</tr>
<tr>
<td>1.</td>
<td>0.014 (2150)</td>
<td>0.070</td>
</tr>
<tr>
<td>2.</td>
<td>0.024 (2250)</td>
<td>0.048</td>
</tr>
</tbody>
</table>

Note: Figures in brackets denote sample length (μm).

Machining grooves or "tracks" persist even in this fine regime of surface finish (Figure 6.31c). In estimation of the surface roughness they can have a major influence (Table 6.6). Local surfaces are generally smooth, and disregarding the machining tracks the CLA roughness of the 2 bars is 0.003 microns and 0.012 microns, far lower than the nominal value of 0.05 microns as given by P S Marsden Ltd. Taking the grooves into account the CLA roughness increases to 0.01 and 0.02 microns, but is still lower than 0.05 microns. It is possible that either the instrument used by P S Marsden Precision Engineering Ltd for measuring surface roughness is not sensitive enough at this fine level and a rms value is derived under false criteria, or there is error bias by the operator. This is supported by the fact that the maximum peak to valley heights recorded from the tracks are approximately 0.04 and 0.07 microns, and 0.05 microns falls within this range.
An inhomogeneity is seen in the surface profiles where the left side is smoother than the right for both bars (Figures 6.31a, 6.31b). This is probably due to the diamond wheel bearing an uneven load on the surface of the bar, causing greater roughness at the region of highest contact load.

The position of the tracks is independent of this feature as they have been formed by the previous grinding stage (350 diamond grit wheel). The tracks do not cover the whole surface of the bar but exit only in certain areas. This means that either the fine machining stage did not polish the surface uniformly, or more probably the depth of damage created by the previous grinding stage varied in different areas.

6.3.2 Optical and SEM Examination

The presence of "remnant tracks" (Figure 6.32a-c) is evidence of some form of damage left over from the coarse machining stage. Examining the tracks at 0 deg tilt, using secondary electron imaging it can be seen the damage manifests itself in the form of a combination of intergranular phase and grain pull-out (Figures 6.33a-c). The large feature seen in Figure 6.33a is revealed in Figure 6.33c to be the site of a large grain which has been pulled-out.

The tracks have a finite length of up to 1.5 mm and a width of up to 16 microns. This is further evidence that the deformation caused by diamond grinding is a combination of axisymmetric indentation and linear single point machining damage mechanisms.

6.3.3 Cross-Section Examination of Fine Machined Surface

6.3.3.1 Optical and SEM Examination

Cross-sectional specimens were produced using the process detailed in Section 5.2.1

No artefacts at the machined surface were identified with the optical microscope using penumbra illumination. A detailed view of the flat machined surface can be seen in Figure 6.34.

6.3.4 TEM Examination

A search was made around the transparent areas of the specimen. However the size of most grains gave rise to an
Figure 6.32a Reflection optical micrograph mosaic of longitudinally machined-bar with random distribution of machining tracks of finite length. 0° tilt

Figure 6.32b The remnant track has a finite width and is seen as surface material pull-out. 75° tilt

Figure 6.32c Magnified view of figure 6.32b, showing pull-out. 75° tilt
Figure 6.33 Machining direction for all three figures

Figure 6.33 Backscattered electron micrographs of surface material pull-out at 0° tilt, which exists in the form of intergranular phase and grain pull-out.
Figure 6.34a Secondary electron image. 3° tilt

Figure 6.34b Backscattered electron image. 3° tilt

Figure 6.34 SEM cross-section view of fine machined surface
overlapping of the microdiffraction patterns. Only a few of the largest grains were analysed under the two beam condition and none revealed the presence of dislocations.

Thus a random search in the bright field imaging mode was carried out over most of the electron transparent areas. Again, no dislocations were detected (Figure 6.35a). Although it appears that little of no plastic deformation is present, it must also be taken into account that due to the nature of machining damage in ceramics, grains in certain orientations suffer little or no deformation. A most likely area of dislocation deformation is around and beneath the remnant machining tracks. However their position location is lost during the specimen preparation process. Microdiffraction patterns were also analysed for evidence of arcing of the diffraction spots, a phenomenon proven successfully used in the highly deformed rough ground surface. Figure 6.35b shows a typical clear diffraction pattern from a hexagonal basal reflection. No arcing is present in this microdiffraction pattern and none was detected in patterns from other grains.

Although it appears that mechanical deformation has been minimised at this level of surface finish it must be noted that tensile stresses of the order of 10–30 MPa transverse and 2–10 MPa longitudinal were measured in the fine machined surface by X-ray microdiffraction analysis (Flewitt 1989).

6.4: THE EFFECTS OF MECHANICALLY POLISHING THE MACHINED SURFACES AND A SURFACE CONTAINING AN INDENTATION

Polishing the coarse machined surface produced the same remnant tracks as were found in the fine machined surface (Figure 6.36)(See Sections 6.3.1c, 6.3.2 and Figures 6.32, 6.33 for fine machined surface). After 1.5 microns are removed, the deeper grooves from the rough surface are still present (Figure 6.36b). At a depth of 2.0 microns a random scattering of tracks from the deepest grooves still exist (Figure 6.36c). At 5.5 microns most tracks have disappeared leaving just two well defined ones (Figure 6.36e), and by 8.5 microns these tracks are very faint (Figure 6.36f). Finally no evidence of enhanced pull-out is present at approximately 9.0 microns depth.
Figure 6.35a Bright field TEM image of large θ-grain in microstructure

Figure 6.35b Microdiffraction pattern of a θ-grain from a hexagonal basal plane
Figure 6.36  Reflection optical micrographs of remnant tracks in the coarse machined surface at increasingly deeper levels in the surface. 0° tilt
Figure 6.36
The indentation cavity is polished away until only the pull-out in the deformed zone is present
With the intermediate machined surface only a few traces remained after 4.5 microns were removed, and at a depth of 6.0 - 7.0 microns all evidence of tracks had disappeared. The tracks on the fine machined surface tended to fade at a depth of around 5.0 microns and a few persistent ones remained to a depth of 6 - 6.5 microns.

The effect of polishing an indented surface can be seen in Figure 6.37 where 2.5μm of material was removed from a polished surface containing a 20kg indentation, giving rise to a "band" around the indentation site. In Figures 6.36 a - n the layer by layer removal of the coarse machined surface containing an indentation is shown. After removing 1.5μm from the surface the indentation contains a "band" around the indentation site or cavity. Further polishing to 2.0μm, 3.0μm, 5.5μm and 8.5μm depths naturally reduces the depth and also the width of the indentation cavity, but a square band of roughly the same size as at 1.5μm depth is still present. The formation of the band is similar to the formation of remnant tracks under the coarse machined surface. Here the plastically deformed zone around and below the indentation site also contains microcracks which preferentially run along grain boundaries. The mechanical fine polishing interacts with this zone causing pull-out. The indentation cavity reaches a depth of between 8.5μm and 12μm (Figures 6.36f and 6.36g). The pull-out zone extends below this and maintains a square shape to a depth of 17.5μm, approximately 7 - 8μm below the bottom tip of the indentation cavity (Figure 6.36i). Thereafter the pull-out region continues as a circular zone (Figures 6.36j - n).

At all depths the radial/median cracks extend from the edge of the pull-out zone. This lends support to the theory proposed by Chiang, Marshall and Evans (1982) that the median or radial cracks initiated on loading, extend on unloading but are soon terminated within the plastic zone.

Another interesting feature is the small radial cracks which accompany the main radial/median cracks. A small number are present at the surface, but at levels deeper than 8.5μm the number increases, and some are longer with increasing depth. This indicates that while surface flaws may or may not have
Figure 6.37 Polished surface with 20kg indentation, polished down to a depth of 2.5μm. The mechanical polishing causes pull-out within the weakened deformed zone.
initiated the large and small surface median/radial cracks, the deeper ones were certainly initiated by sub-surface flaws creating limited median cracks which did not completely grow on unloading the indentor. It is difficult to ascertain how the main radial/median cracks were initiated. While the machined surface may present an ample population of flaws for the initiation of surface radial cracks under a tensile stress, the high surface biaxial compressive stresses of the order of 140 MPa and 200 MPa may suppress their formation and growth.

The latter has been shown to be true in work carried out by Holm (1989) on the coarse machined material. A series of indentations orthogonal to the machined direction was implemented on the specimen surface. Radial/median cracks parallel to the machining direction were found to be shorter and this is due to the constraining higher compressive residual stress field normal to the machining direction.

6.5 EFFECT OF INDENTING A COARSE MACHINED SURFACE

20kg and 50kg load indentations were made at a distance from one another, but during the 70kg indentation, the specimen splintered apart. This happened within the first seconds in the indentation process and therefore the fracture had occurred during the loading cycle.

In Figure 6.38, by lining together the two fracture halves of the specimen, it can be seen that the line of fracture lies parallel to the machining direction. This is expected as median semi-elliptical sub-surface machining-induced flaws which lie in this direction are opened up by the tensile loading force. Furthermore it can be seen that the median/radial crack from the 70kg load indentation propagated and "sensed" the tensile field present around the 20 kg indentation site and linked up with the radial/median crack of that indentation. Further propagation of the crack resulted with it linking up with the radial/median crack of the 50kg indentation. The two fracture halves were also viewed in cross-section using scanning electron and optical microscopy. As the indentation sites were cleaved exactly along the radial/median cracks, the indentation sites could be clearly observed. In Figure 6.39 the semi-circular plastically deformed zone can be observed under the 20kg indentation site with a...
Figure 6.38 Optical micrograph mosaic of bird's-eye view of indented machined surface showing link-up of fracture path and radial/median cracks.
Figure 6.39 SEM cross-sectional view of fracture surfaces of the cleaved 20Kg indentation site. Arrows indicate the boundary of the deformed zone.
median crack under the deformed zone tip. Two lateral cracks on each side of the deformed zone are also present, however they do not originate from the tip of the plastically deformed zone, but halfway up. This may be due to the influence of a greater stress field mismatch at the machined plastic/elastic deformed layer boundary. The 50kg indentation site also shows the same deformation/crack characteristics (Figure 6.40). Examining the 70kg indentation site (Figure 6.41a) the striations in the fracture surface A are all aligned in the direction of the indentation site and this confirms that this was the origin of fracture. On the right hand side of the deformed zone a straight lateral crack points upwards to the surface (Figure 6.41c). In Figure 6.41b, which shows the opposite fracture surface, the lateral crack can be seen curling up towards the surface, typical of heavily loaded specimens.

6.6 IMPURITY FRACTURE ORIGIN OF THREE POINT FLEXURAL RUPTURE TEST

Within seconds of applying the load the bar fractured at a load registered by the load cell as 44.3% of 2kN. Using the equation as given by Stanley et al (1976) the nominal strength \( \sigma \) is:

\[
\sigma = \frac{3Wl}{2bd^2}
\]

where
\[
W = \text{load at point of fracture}, \ 90.3\text{kg}
\]
\[
l = \text{span}, \ 9.998\text{mm}
\]
\[
b = \text{width of bar}, \ 3\text{mm}
\]
\[
d = \text{thickness of bar}, \ 3\text{mm}
\]

Thus \( \sigma = 50 \text{ MPa} \)

The M.O.R. values of the longitudinally ground coarse machined surface measured by Mr R Quinn (1989) at Rolls-Royce Leauesden fell in the range 983MPa to 590MPa, excluding the 10% of bars that failed due to the presence of processing flaws. The value of 50 MPa obtained is very low and a non-machining defect was considered to be the most probable cause of failure.

A SEM cross-section examination was made of the remaining fracture surface (Figure 6.42a) (the other one had been lost in
Figure 6.40 SEM cross-section views of 50Kg indentation site
Figure 6.42a
SEM cross-sectional view of bar fracture surface

Figure 6.42b
SEM cross-section view of fracture surface of top part of bar which has undergone the tensile stress. Fracture lines indicate the origin of fracture which is at the point marked "0" 130µm below the machined surface. 43° tilt
a fragmented wedge chip form). There is much fracture detail but the surface texture indicates an area of interest in the top right hand corner 130μm below the machined surface (Figure 6.42b). Here, it is clear from the fracture path lines that the origin of fracture is at the centre of the area marked "o" on the micrograph, near the sharp step in the fracture surface. Examining the centre of this area in detail (Figure 6.43) the origin of failure is shown to be due to a "black winding" processing flaw approximately 100μm in length.

Using a JEOL 35 CF electron microscope the elemental composition of the processing flaw was analysed by energy dispersive X-ray spot analysis, using a 15kV electron beam. Traces of aluminium and chlorine were found (Figure 6.44). These were not expected as the impurity concentration of aluminum and chlorine in the starting powder was 22 ppm and 50 ppm respectively (see UBE SN E10 powder specification Section 2.3.2, Table 2.1) and these should not be sufficient to concentrate in one area thereby forming a potential region for failure. It is possible, and more likely therefore, that while Turner and Newall changed to a silicon nitride ceramic ball mill rather than using a steel one to avoid contamination (see Section 3.3.2), the type used was probably 102A2 sintered reaction bonded silicon nitride which contains 10 wt% Y₂O₃, 2wt% Al and 2 wt% Cr sintering additives, and is fabricated by Turner and Newall. Traces of Mg and Ca can not be explained.

In conclusion this serves to illustrate the importance of statistics and probability in the failure of brittle ceramics. While 90% of bars tested by Mr R. Quinn (1989) at Rolls-Royce Leavesden failed due to machining induced flaws, the only bar tested by me failed due to a processing flaw.

6.7 SINGLE POINT SCRATCH TESTS

6.7.1 Morphology of Grooves and Material Fragmentation

25gm: The indentor has left only a thin line of deformation (Figure 6.45a) which exists in the form of grain boundary fracture (Figure 6.45b, 6.45c). Some material debris is present as 0.25μm particles.

50gm: A groove shape begins to form at this load (Figure
Figure 6.43 High magnification views of processing flaw
Figure 6.44 Energy dispersive X-ray spot analysis of processing flaw showing traces of Al and Cl
Note:
For all the scratch test results the direction of scratching will be indicated by an arrow by the side of the figures.
6.46a). The deformation is not symmetrical, where on the left hand side of the groove there is grain boundary fracture, and on the right hand side lies material debris which has separated from the original surface (Figures 6.46b, c). The debris exists as fragments of material up to 3 or 4\(\mu\)m in size, and the structure of the fragment in Figure 6.46c suggests that it originated from within the groove site.

100gm: It was found that the Vickers diamond pyramid indentor was slightly worn (Figure 6.47) and the effect of the unsymmetrical rounded tip can be seen in the form of a shallow "furrow" which exists at the centre of the groove site (Figure 6.48a). The furrow consists of grain boundary fracture and pull-out, and the larger material fragments are still displaced to the right hand side of the groove (Figures 6.48a, b). On the left hand side of the groove there appears to be only grain boundary fracture. The number and size of debris fragments is slightly higher than for the 50gm load, and their structure suggests that whole groups of grains are detached from the surface (Figure 6.48c).

200gm: The groove continues as a slightly deeper furrow, but the material debris is present in two forms (Figures 6.49a, b, c). Some pieces up to 20\(\mu\)m in size appear as solid fragments as for the previous loads, while others have a "flaky" structure, and are probably formed by a different removal mechanism.

500gm: A significant change in the morphology of the groove and debris is reached at this load. Large fragments ranging from 10 to 70\(\mu\)m in size are left at regular intervals along the whole length of the groove (Figures 6.50a, b, c). The fragment structure at high magnification can be seen to be of highly deformed and compacted "platelets" of material (Figure 6.51a), which suggests that their formation derives from a build up of debris under the diamond tip, which is finally pushed to the side. This is supported by the fact that there is no evidence of large scale separation along the groove. Within the groove there are areas which contain scale-like cracks which "step-up" in the scratching direction (Figures 6.51b, c). These are similar to "chatter" marks that are common on surfaces which have been ground with a non-stiff machine, where a stick-slip
Figure 6.47a Vickers diamond pyramid indentor tip is worn.
45° tilt

Figure 6.47b High tilt view. 77° tilt
movement causes vibration in the grinding process. During the tests a high pitch scratch noise was heard which was similar to a piece of chalk being scratched across a blackboard. With a traverse speed of roughly 1 cms\(^{-1}\) and an average crack separation of 2.2\(\mu\)m, the chatter marks represent an audio frequency of 4.5 kHz, and this is approximately consistent with the noise heard.

**750gm:** The nature of the material deformation is the same as for the 500gm load (Figures 6.52a, b, c, 6.53a). The larger fragments are up to 200\(\mu\)m in size and the chatter marks represent an audio frequency of approximately 3.8 kHz, having an average separation of 2.6\(\mu\)m (Figure 6.53b,c). An extra interesting feature in Figure 6.52a is the presence of two depressions in the groove each accompanied by a crack lying across the groove. This suggests that there was some form of weakness underneath these points which caused the groove to collapse.

**1000gm:** A further change in the deformation process occurs at this higher load, where chevron cracks are formed along the sides of the groove, and the brittle fracture material removal process is enhanced via "small" and "large-scale" chipping. A distinction between the two chip types will be taken according to the geometry of the fracture process. "Small" chips occur individually along one side of the groove only, whereas "large" chips have two facing fracture surfaces on either side of the groove, deriving from one critical fracture event. Measurements of crack and chip sizes and angle of inclination to the groove direction are described in Figure 6.54 and are entered in Tables 6.7, 6.8.

Chevron cracks were formed randomly along both sides of the groove length, and in some areas a symmetry in the crack formation occurred (Figure 6.55a). The cracks are inclined in the direction of scratch motion (Figure 6.55b) and illustrate the effect of traversing a Vickers diamond indentor; the change in the dynamics of the deformation-fracture process results in distorted radial cracks, as compared to a quasi-static indentation process. Back scattered electron analysis of the crack in Figure 6.55c shows that the mode of crack propagation is via an intergranular route. The crack lengths ranged from 4.4\(\mu\)m to 65.7\(\mu\)m and the angle of inclination to the groove θ
Figure 6.54 Chevron crack and chip parameters
ranged from 26.3° to 90°. It was found that the smallest cracks tend to be at wide angles to the groove (4.4µm = 90°, 5.8µm = 65.0°, 7.3µm = 68.2°) whereas the largest cracks lie at small angles (65.7µm = 20.2°, 50.4µm = 28.4°, 49.6µm = 26.5°). The large scatter in the crack lengths and angles derives from a scratching process that contains a significant contribution from vibrations in the non-stiff machine.

The small chips are randomly distributed along the groove length and range from 8.0µm to 26.3µm in width and 19.0µm to 54.0µm in length. Roughly 50% are irregular in shape and do not approximate to a semi-elliptical curve. The angle of the "leading edge" to the groove is small and ranges from 20.7° to 46.2°. This is consistent with the measurements of the chevron cracks, where the longest cracks most likely to cause chipping lie at angles of approximately 20-30° to the groove.

Large chips, also randomly distributed along the length of the groove, occur in regions where a critical tensile stress enforced an extension of chevron and lateral cracks, causing brittle fracture and lift-out of chips from the surface (Figure 6.56a). The leading edges of the large chips extend to a much greater length than the longest chevron cracks, and this may be due to a process whereby crack propagation in the weakest crack areas leads to fracture and chip removal. However, the leading edge lengths fall in a wide range from 31.2µm to 104.0µm while the angles to the groove are consistently low ranging from 22.8° - 48.9°. This shows that there exists a critical low angle at which cracks develop and propagate causing fracture, and this is determined by the dynamics of the scratch process; principally the indenter size and shape, the load applied and the speed of traverse. A piece of material debris can be seen to lie in the left hand side fracture area in the chip shown in Figure 6.56a. Examination at high tilt shows that it has a bulk shape and does not appear to be compatible with the morphology of the fracture area (Figures 6.56b, c). However, it is possible that the separated fragment may have "curled up" due to high local residual stresses, this being similar to the severe buckling caused in a thinned foil specimen of the coarse machined surface (Section 6.1.4.5). The chip in Figures 6.57a, b may provide an alternative argument, however. The left hand side fracture piece
Figure 6.56c 75° tilt

Figure 6.56a 0° tilt

Figure 6.56b 75° tilt

Figure 6.56 1000gm load scratch
has not left the surface and exists in the same form as a "flat platelet". In the right hand side fracture area though lies another fracture piece with a bulky form, and this may have replaced the platelet lying further away on the free surface. This could happen if a build up of material debris under the indentor tip is released by the change in scratch conditions when a large chip is formed, and is thus deposited in the chip fracture area. It is noted though, that most of the material debris lies at a distance from the scratch area (not encountered at previous loads), and suggests that a high energy "splinter" material removal process occurs along the whole length of the groove. The chip fracture areas rarely exist in the form of smooth "craters" but have either irregular surfaces, or sometimes a double "bowl" structure, and this reveals a complicated and variable fracture process in the link up of chevron and lateral cracks, which is further enhanced by the low stiffness in the scratch machine.

All large chips are accompanied by depressions in the 4.4μm groove depth, and these extend down to depths of 8.2μm; associated with the depressions are large cracks which run across the groove width (Figures 6.56a, b and 6.57a, b). No depressions were found to exist below the level of the chip fracture surfaces, and this suggests that the groove bottom collapsed after the two chip areas were lifted out. However it is also a lesser possibility that the indentor tip momentarily slowed down during the chip-fracture process and thus the enhanced dead load condition created a deeper impression. The slow down in the indentor may have attributed to the build up of material debris under the indentor tip as reasoned above in the explanation of bulky fracture pieces existent in the fracture chip areas. This is further supported by the groove in Figure 6.52a made by a 750gm load. No chipping is present but a large fracture piece lies along the side of an area of the groove where there are two depressions. A build up of material debris under the indentor tip and the sudden release of a compressed fracture piece may have caused the tip to slow down without creating chevron cracks and chip-fracture, but causing a depression in the groove depth.

A close examination of the scale-like marks shows that small
cracks exist within the larger "layered" cracks (Figure 6.57c). The depth of the larger cracks has not been ascertained but there is a hint of evidence in Figure 6.55b that there is a link up between these and the chevron cracks. The separation of the scale-like marks at this higher load was measured to be 4.6 μm, and this represents an audio frequency of 2.2 kHz.

1500gm: In general the longer chevron cracks remain at low angles to the groove (48.2 μm = 28.3°, 39.4 μm = 38.8°, 29.2 μm = 39.5°), while the shorter cracks lie at wide angles to the groove (7.3 μm = 43.1°, 8.8 μm = 56.1°, 8.8 μm = 78.9°). However the mean angle has slightly increased even though the cracks do not appear to have increased in length (Table 6.7). This is probably due to the fact that a greater number of lower angle chevron cracks are "converted" into chips at this load, therefore leaving a higher number of wide angle cracks.

Although small-scale chipping is still present along the groove edges (Figure 6.58a), material removal is dominated by large-scale chipping. The large chips are greater in length and width as compared to the ones produced by a 1000gm load, and still subtend at low angles to the groove. The chip site shown in Figure 6.58b contains a large but wide angle chevron crack that failed to propagate and link with the lateral cracks and cause large scale chipping. A low angle view of the chip (Figure 6.58c) reveals how chips are lifted out of the free surface in the form of platelets, which in this case have not completely left the fracture area.

The depressions present at chip sites reach levels of 8.2 μm to 12.1 μm below the mean 7.6 μm groove depth, and as for the 1000gm load no depressions are found to descend below the chip fracture areas. The depression in the centre of the symmetrical chip in Figure 6.59a contains a well-defined crack across the groove width. A low angle view of the chip site confirms the process of groove fracture, where the groove bottom collapses on top of the chip fracture area (Figure 6.59b).

Material fragments which are left by the side of the groove retain the highly deformed compacted structure (Figure 6.59c), and are still accompanied by a sub-micron powder of debris.
Areas which contain chatter marks are still randomly distributed on the groove bottom and do not specifically precede or follow any large-scale chipping or concentrated chevron crack areas. The chatter mark separation of 3.5μm represents an audio frequency of 2.9 kHz.
<table>
<thead>
<tr>
<th>LOAD P (g)</th>
<th>LENGTH (µm)</th>
<th>Cc (µm)</th>
<th>ANGLE</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>RANGE</td>
<td>AVERAGE</td>
<td>RANGE</td>
</tr>
<tr>
<td>1000</td>
<td>4.4 - 65.7</td>
<td>19.9 (± σ = 16)</td>
<td>26.3° - 90.0°</td>
</tr>
<tr>
<td>1500</td>
<td>7.3 - 48.2</td>
<td>17.5 (± σ = 10)</td>
<td>28.3° - 90.0°</td>
</tr>
<tr>
<td>2000</td>
<td>13.0 - 98.8</td>
<td>43.8 (± σ = 26)</td>
<td>37.8° - 90.0°</td>
</tr>
<tr>
<td>LOAD P (gm)</td>
<td>LEADING EDGE LENGTH ( (\mu m) ) ( L_E )</td>
<td>WIDTH, ( W_L ) ( (\mu m) )</td>
<td>LENGTH, ( L_L ) ( (\mu m) )</td>
</tr>
<tr>
<td>-------------</td>
<td>---------------------------------</td>
<td>-----------------</td>
<td>-----------------</td>
</tr>
<tr>
<td>1000</td>
<td>RANGE</td>
<td>AVERAGE</td>
<td>RANGE</td>
</tr>
<tr>
<td></td>
<td>31.2-104.0</td>
<td>54.6(( \pm \sigma = 23 ))</td>
<td>21.6-53.8</td>
</tr>
<tr>
<td>1500</td>
<td>26.0-93.6</td>
<td>57.7(( \pm \sigma = 19 ))</td>
<td>23.0-59.4</td>
</tr>
<tr>
<td>2000</td>
<td>59.8-104.0</td>
<td>72.8(( \pm \sigma = 15 ))</td>
<td>28.6-53.3</td>
</tr>
<tr>
<td></td>
<td>22.8-48.9</td>
<td>36.1(( \pm \sigma = 9 ))</td>
<td>28.9-62.1</td>
</tr>
</tbody>
</table>
2000gm: The features present in the 1500gm load groove are repeated at 2000 gm, although the general level of deformation and fracture has increased; the chevron crack and chip sizes are larger and a greater volume of small material debris is present around the scratch area (Figure 6.60a, b, c). Also, no chip platelets are left in the fracture areas due to the high fracture energies involved. Large chips in the 1500 gm groove may have been followed by a fracture event at a distance of 40-60µm further along the groove (Figure 6.58b, 6.59a), but the deformation-fracture at 2000gm is so concentrated that a new fracture event may occur as part of the preceding chip site (Figure 6.60a).

The depressions at the chip sites reach levels of 8.9 to 11.5µm, below the mean groove depth of 7.9µm, and as for the large-scale chipping created by the previous two loads, the chip fracture areas are rarely smooth "craters". The shape of the groove left by the rounded indentor tip can be seen to take a symmetrical form under this heavy load. The chatter marks have not changed in form and the separation of 2.3µm, giving an audio frequency of 4.4 kHz, is still consistent with the results from the previous four loads.

6.7.2 The Diamond Vickers Indentor

It was found that the indentor tip was slightly worn (Figure 6.47). Examination from a different angle reveals the rounded tip and also damage in the form of cracking and chipping (Figure 6.61a). The cracking is probably a result of tangential and frictional stresses causing cleaving during previous scratch processes.

After scratching had taken place from the 25gm load to the 1000gm load the indentor tip was found to be covered with material debris (Figure 6.61b) which probably adheres to the diamond surface via attractive electrostatic forces. The debris is the same as that found around the groove sites and is a combination of small particles, platelets, flakes and comminuted, compacted fragments (Figures 6.61c, 6.62a, 6.62b). At a high tilt it can be seen that the diamond tip is not only rounded due to wear, but it also contains highly compacted material debris which was found
to be impossible to remove with extensive and vigorous cleaning with acetone, and methanol in an ultrasonic bath. Further scratching with loads of 1500 and 2000 gm caused an increased compaction of material on the diamond tip as well as further cleaving (Figure 6.62c). The fragment lying on the tip is a section of diamond material which has been separated from the area on the right hand side face.

6.7.3 Sub-Surface Median Cracks
The grooves were viewed in normal cross-section using penumbra illumination, and sub-surface median cracks were found to exist under all scratches, except for the 25gm and 50gm load scratches. The largest crack measured for each load is entered in Table 6.9 (Note: a suitable cross-section cut for the 2000gm load was not achieved).

<table>
<thead>
<tr>
<th>LOAD (gm)</th>
<th>CRACK DEPTH (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>14.2</td>
</tr>
<tr>
<td>200</td>
<td>19.9</td>
</tr>
<tr>
<td>500</td>
<td>36.2</td>
</tr>
<tr>
<td>750</td>
<td>41.0</td>
</tr>
<tr>
<td>1000</td>
<td>82.8</td>
</tr>
<tr>
<td>1500</td>
<td>105.0</td>
</tr>
</tbody>
</table>

Typical cracks only from the highest load grooves were photographed as great difficulty was found in trying to illuminate the area correctly with the penumbra illumination method, and the largest cracks would facilitate the technique. Highly magnified prints of the negatives were made as
considerable manipulation in lighting conditions and shadowing was needed to highlight and maximise the contrast of the crack, and a larger useable area would help this process.

The morphology of the cracks is similar to those found under the 0.4μm CLA rough ground surface. A typical crack in Figure 6.63 initiates at the plastic/elastic boundary of a semi-circular deformed zone under the groove. Evidence of microcracks in this plastically deformed zone is also present. Similarly the crack is not straight, but here curved to the left. In Figures 6.64 and 6.65 multi-cracks initiate along the periphery of the semi-circular deformed zone similar to the multi-cracks found under the deep grooves in the rough ground surface. This is further evidence that protruding diamond particles in a diamond grinding wheel cause deformation-fracture similar to a single point scratch, and the residual stress left in a ground surface is due to an accumulation of multi-particle contact events.
CHAPTER 7. DISCUSSION

SUMMARY

This chapter provides a discussion on all the results obtained from the analysis of the coarse, intermediate and fine machined surfaces. A description of machining damage includes and ties in the results from scratch tests and the effects of polishing indented and coarse machined surfaces, together with brittle material fracture theory and work carried out by other authors in this field. The discussions closely take account of the original machining and scratching conditions.

The discussion also covers the fracture strength work carried out at Rolls-Royce by R Quinn, and X-ray diffraction residual stress measurements carried out at the CEGB by P E J Flewitt. It shows that their results are consistent with the nature and depth of machining damage identified at the University of Surrey.
CHAPTER 7. DISCUSSION

7.1 COARSE MACHINED SURFACE

In the study of strength controlling defects in a brittle material it is important to characterise the weakest link as this can be an initiator for brittle failure due to fracture (Sedlacek 1972, Marshall and Lawn 1980, Kirchner and Isaacson 1982 and 1983, Marshall et al 1983, Johnson-Walls et al 1986, Govila 1988, Quinn 1989). This is apparent in the mechanical damage created in the surface and sub-surface region due to grinding. A surface which has been diamond ground in certain specified conditions may be said to contain a general level of sub-surface damage. As described in Section 4.4 machining damage is an accumulation of a large number of isolated sharp particle contact events (Marshall et al 1983), where each elastic/plastic contact causes irreversible damage. Therefore regions with higher surface roughness contain greater levels of sub-surface damage than other regions. However, it is the deepest grooves caused by enhanced particle contact events, that constitute areas of greatest deformation, although it has been noticed that the deepest grooves do not necessarily occur in the regions of greatest roughness.

Machining Grooves and Material Pile-up

The force of an individual diamond within the grinding wheel should be independent of the depth of cut, and the total force on a grinding wheel is believed to increase with the depth of cut, as presumably more diamonds are involved in the machining process (Johnson-Walls et al 1984). However, if one particular diamond in the upper range of the permitted mean size protrudes from the surface, even for one revolution, then its effect is magnified (Zhang et al 1988). A profile analysis of the coarse machined surface has shown the existence of well-defined grooves of \( \approx 2 \, \mu m \) depth.

Close examination reveals that pile-up around a groove is caused by the action of plastic flow, where the volume of the groove created by a singularly large diamond is accommodated by the surrounding material. The shape of the groove may vary and will depend on the shape of the diamond particle.
Sub-Surface Median Cracks

Cracks normal to the machined surface were identified from normal cross-sections under the optical microscope using penumbra illumination. They are made visible due to the penetration of light which illuminates sub-surface layers of the ceramic which has a degree of optical transparency. The position of the cracks is consistent with machining-induced sub-surface deformation and median cracks that are formed under the most severe grinding grooves and they extend from 6µm to 45µm from the machined surface with an average separation of 227µm normal to the machining direction. The cracks initiate at an average depth of approximately 4-5µm below the machined surface at the plastic/elastic boundary, and this can be taken as the average depth of the layer of residual compression, as many cracks do not extend from under deep grooves, but from flat or even slightly peaked surfaces. This is due to deep grooves being removed on further machining which leaves behind just the median crack. Further evidence of the depth of the residual compressive layer is seen in the average 4.0µm band of mechanical damage in a polished 7°oblique section of the machined surface (Quinn 1989). The median cracks do not extend directly normal to the surface and 61% are deviated at an angle of 5° or more. This is either due to the shape of the diamond particle, which creates an unsymmetrical groove and consequently an unsymmetrical subsurface deformation, or local stresses that interact with the major damage site. SEM examination of the cross-sections shows that the cracks only break the specimen surface intermittently. This interesting feature is certainly due to compressive stresses being formed in the specimen surface as a result of mechanical polishing which tend to close the crack surface. A delta tributary system of microcracks converges from the machined surface to the start of the crack and it is believed that either they may be precursors to crack initiation, or they are kept open by the short range local residual tensile component acting under the deformed zone.

Parallel cross-sections of the machined surface revealed the subsurface median cracks to be semi-elliptical in shape along the machining direction. They were found to be 19µm - 100µm long, with an average separation of 413µm. Their shape suggests that loading during machining causes a combination of axisymmetric
indentation damage and linear deformation-fracture. Directionality due to the machining direction may also be seen in the distortion of some semi-elliptical cracks. Measurements of cracks from the normal and parallel cross-sections also provides an estimation of the concentration of cracks in the machined surface, and this was found to be approximately 18/cm². Backscattered electron analysis of the median cracks viewed normal to the machining direction reveals that the mode of fracture is primarily intergranular with some transgranular fracture present. This is not consistent with some observations of ceramic brittle fracture in flexural rupture tests carried out at room temperature, where the mode of crack propagation was primarily transgranular due to fast fracture (Govila 1985 and 1988); (he found that due to the very high strain rate in the vicinity of the crack tip the crack found a transgranular path easier than a more deformation-accommodating tortuous grain boundary one). Flexural rupture tests create unstable crack propagation, whereas cracks formed during machining may be more stable with a slower growth, where the crack propagates by plastic separation of the grain boundary crystalline intergranular phase.

**Tem Analysis of Machined Surface Layer**

TEM examination of normal cross-sections of the machined surface has shown evidence of mechanical deformation. The concentration of damage and lattice strain at the machined surface is so high that few dislocations are individually resolvable while the dislocation density is estimated to be approximately in the order 4 x 10¹⁴ m⁻². Electron microdiffraction patterns are distorted by the deformation which causes streaking of the diffraction spots. This effect tends to disappear at a depth of 4-5μm at the plastic/elastic boundary, and this is further evidence of the existence of a uniform layer of residual compression. The depth of 4-5μm is also consistent with the average crack initiation depth of 4-5μm. Local stress concentrations occur under the largest damage sites, and this has been seen in median cracks that initiate at depths down to 8.7 μm, which is below the layer of residual compression.

**Fracture Strengths**

Fracture strengths of transversely machined bars may be
predicted by applying measured crack sizes to the expression \( A\sigma c_0^{1/2} = K_{IC} \) (See Section 6.1.6). The predicted fracture strength of \( \sigma = 540 \, \text{MPa} \) derived from the coarse machined surface using the measured crack size of 45.7\( \mu \text{m} \) can be tentatively compared to experimental results, and in fact appears to be consistent with the results obtained by Quinn (1989) in Table 7.1 where the fracture strengths for the coarse transversely machined bars fell in the range 402 - 592 MPa. The mean strength using the same expression translates to crack depth of 51.2\( \mu \text{m} \).
TABLE 7.1: MODULUS OF RUPTURE (R) VALUES FOR THE COARSE, INTERMEDIATE AND FINE MACHINED BARS


<table>
<thead>
<tr>
<th>Surface Finish</th>
<th>Range (MPa)</th>
<th>R&lt;sub&gt;mean&lt;/sub&gt; (MPa)</th>
<th>W&lt;sub&gt;M&lt;/sub&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coarse</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.4μm L</td>
<td>590-982</td>
<td>764.4</td>
<td>9.97</td>
</tr>
<tr>
<td>0.4μm T</td>
<td>402-592</td>
<td>508</td>
<td>10.92</td>
</tr>
<tr>
<td>Intermediate</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.1μm L</td>
<td>797-1068</td>
<td>960.6</td>
<td>14.42</td>
</tr>
<tr>
<td>0.1μm T</td>
<td>544-770</td>
<td>654.2</td>
<td>10.09</td>
</tr>
<tr>
<td>Fine</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.05μm L</td>
<td>851-1022</td>
<td>917.2</td>
<td>18.12</td>
</tr>
<tr>
<td>0.05μm T</td>
<td>645-838</td>
<td>729.3</td>
<td>13.88</td>
</tr>
</tbody>
</table>

where L = longitudinally machined
T = transversely machined
R = modulus of rupture
W<sub>M</sub> = Weibull modulus

The above expression does not take into account the tensile residual component acting on a sub-surface median crack. By accommodating stable crack growth during failure in the expression, using C<sub>m</sub> = 5 derived by Marshall et al (1983) from a series of machined and indented surfaces, the predicted fracture strength σ = 240 MPa. This figure appears to be low due to little or no crack extension before failure, and a compensating effect due to the residual compressive layer of the order of 200 MPa (Flewitt 1989) acting normal to the machining direction.

The effect of surface finish on the strength of a ceramic is
illustrated in Table 7.1 where the mean strength and Weibull modulus are seen to increase from the coarse machined surface to the fine machined surface. The increase is significant and expected considering the surface placed under a tensile stress ranges from one containing high mechanical deformation (Figure 6.2, 6.5, 6.6), deep grooves (Figure 6.1a,b) and large sub-surface cracks (Figure 6.9), to one which is highly polished (Figures 6.31a,b) with no apparent grooves or sub-surface cracks (Figure 6.34).

It must be noted though that the machined bars came from five billets which gave rise to a variability in strength between billets Table 7.2. The variability is probably due to differences in processing conditions.

<table>
<thead>
<tr>
<th>Billet</th>
<th>Mean (MPa)</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>667.4</td>
<td>52.4</td>
</tr>
<tr>
<td>2</td>
<td>741.2</td>
<td>52.9</td>
</tr>
<tr>
<td>3</td>
<td>761.8</td>
<td>39.5</td>
</tr>
<tr>
<td>4</td>
<td>767.4</td>
<td>20.5</td>
</tr>
<tr>
<td>5</td>
<td>884.9</td>
<td>62.8</td>
</tr>
</tbody>
</table>

The distinct anisotropy of the fracture strengths in the coarse machined surface (Table 7.1) is a direct result of the machining direction. The surface deformation and grooves (Figures 6.1a,b), sub-surface deformation and cracks (Figures 6.9 and 6.23) lie parallel to the machining direction, and these result in lower fracture strengths when subjected to a normal as compared to a parallel tensile stress. Thus the transversely machined bars show lower fracture strengths. Anisotropy in the machining-induced deformation is also seen in residual stresses measured
by Flewitt (1989)(Table 7.3), where material displaced by grinding grooves causes local residual stresses which overlap forming an overall layer of residual compression with the highest stress acting normal to the machining grooves. Measurements of the compressive stress layer taken transverse to the grooves are uniform but those taken parallel show a wide variation. The compressive stress layer must have an underlying compensating tensile field. As the penetration depth of Cr radiation in silicon nitride is approximately 12μm the X-ray strain measurements will have incorporated both the compressive and tensile components therefore giving averaged readings probably lower than the true value.

**TABLE 7.3:** RESIDUAL STRESS MEASUREMENTS OF THE COARSE AND FINE MACHINED SURFACE

Source: Flewitt (1989), CEGB (Now Nuclear Fuels PLC).

<table>
<thead>
<tr>
<th>Position</th>
<th>0.4μm CLA</th>
<th>0.05μm CLA</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Coarse Finish</td>
<td>Fine Finish</td>
</tr>
<tr>
<td></td>
<td>(MPa)</td>
<td>(MPa)</td>
</tr>
<tr>
<td>Longitudinal</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>-32 ± 42</td>
<td>2 ± 10</td>
</tr>
<tr>
<td>2</td>
<td>-107 ± 10</td>
<td>10 ± 10</td>
</tr>
<tr>
<td>3</td>
<td>-155 ± 14</td>
<td>3 ± 8</td>
</tr>
<tr>
<td>Transverse</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>-201 ± 12</td>
<td>29 ± 5</td>
</tr>
<tr>
<td>2</td>
<td>-202 ± 9</td>
<td>25 ± 12</td>
</tr>
<tr>
<td>3</td>
<td>-216 ± 84</td>
<td>10 ± 12</td>
</tr>
</tbody>
</table>

Note: Residual stress values calculated from X-ray strain measurements (Cr Kα radiation) using \( E = 322 \) MPa and \( \nu = 0.268 \).
7.2 **FINE MACHINED SURFACE**

**Fracture Strength**

Anisotropy in the fracture strengths still persists in the fine machined surface bars (Table 7.1) and is consistent with the behaviour of the coarse machined surface, where the transversely machined bars show lower fracture strengths. Therefore, although the mean strength of the bars has increased with a higher quality finish, machining-induced deformation still exists at this level of surface finish and its effect is still anisotropic.

**Machining Schedule**

The machining schedule for producing a fine surface finish starts with 150 and 350 grit diamond wheels, therefore causing the extent of "coarse" damage examined in Section 6.1. Continuing with a 600 grit diamond wheel, (or 400 grit cup wheel) with successive 5.0μm cuts, severe surface and sub-surface deformation and large cracks are greatly reduced and finally completely removed. However even though a smooth polished surface is achieved some form of machining damage is still present. (Note: whenever a bell wheel is used for final finishing any effects on fracture strengths due to directionality in machining damage introduced in previous machining stages may distorted).

**Remnant Tracks**

The presence of "remnant tracks" is evidence of high localised damage underneath previous grinding grooves in the coarse machined surface. Around a groove there is an underlying plastically deformed zone which extends deeper than the overall deformed sub-surface layer. The deformed zone contains microcracks which preferentially run along the grain boundaries, and are enhanced by the short range local tensile stresses acting under the deformed zone. The existence of microcracks is independent of the presence of a median crack (and the tensile field itself also does not depend on the presence of a median crack). After a deep groove is removed the mechanical fine machining interacts with the sub-surface deformed zone, causing pull-out in the areas containing microcracks, thus leaving remnant tracks. These measure up to 1.5mm in length and illustrate that deformation due to machining is a combination of axisymmetric indentation and linear single point machining damage.
mechanisms. The track widths of up to 16µm is consistent with the deep groove widths of up to 18µm in the coarse machined surface and the 15-16µm widths of the tributary system of microcracks seen in cross-sections of the coarse machined surface.

The presence of remnant tracks in the fine machined surface is consistent with the effects of polishing the coarse (Figure 6.35) and intermediate machined surfaces. Once the surface roughness is removed, finite length tracks consisting of grain and intergranular pull-out are left. The effect of polishing a deformed region is further illustrated in the zone of grain pull-out created around an indentation implemented in a rough or polished surface (Figures 6.36, 6.37). The area of grain pull-out exactly matches the expected region of plastic deformation caused by a Vickers diamond pyramid indenter.

**TEM Analysis of Machined Surface**

TEM examinations of the fine machined surface using selected area imaging and microdiffraction analysis did not reveal the presence of mechanical deformation or strain in the crystal grains. It must be considered though that due to the nature of machining damage in ceramics, grains in certain orientations will suffer little or no deformation.

**Residual Stresses**

Nevertheless X-ray strain measurements detect the presence of small residual stresses which show anisotropy (Table 7.3), where the values measured transverse to the machining direction are higher than those measured parallel. This is consistent with the results from the coarse machined surface. However, the residual stresses measured are not compressive but tensile and this can be explained as follows. There exists a compressed surface layer in the fine as well as the coarse machined surface and this must also have an underlying compensating tensile field. The Cr radiation penetrates to a depth far below the deformed compressive layer and thus the tensile field is mainly sensed giving rise to tensile values.

The result of the compressive stress can also be seen in the convex surface curvature across the width of the longitudinally
machined bars in the intermediate as well as fine machined surfaces. Approximating the surface curvature to an arc of a circle the radius of curvature in the intermediate machined surfaces ranged from 0.9 to 1.2m, while the fine machined surface which contains a lower level of compressive stress, the surface curvature was lower and the radius of curvature was measured to be 9 and 12.6m.

The presence of machining tracks and residual stresses in the fine machined surface therefore explain the anisotropic behaviour in flexural rupture tests carried out by Quinn (1989) (Table 7.1).

7.3 SINGLE POINT SCRATCH TESTS

As the Vickers pyramid diamond indentor was slightly worn (Figure 6.47) true scratch parameters such as hardness were untenable from the groove dimensions. However, diamond grinding particles set on the rim of a grinding wheel cannot be said to be exactly sharp, and therefore the experiment was in fact closer to the diamond machining situation, especially when considering the effect of a protruding diamond particle in the wheel on the ceramic material.

The thorough specimen cleaning process carried out before the scratch tests and the careful gold coating afterwards proved to be successful as material debris on the specimen surface was examined with only a limited amount of charging and burning.

Morphology of Grooves and Material Fragmentation

Various stages in the scratch deformation process with increasing load was noted. From 25gm to 100gm loads the material deformation begins as grain boundary fracture with sub-micron particles (Figure 6.45), and continues as a shallow groove or furrow with fragments in the form of groups of grains up to 5μm in size (Figure 6.48), and at 200gm the fragments appear in a solid and "flaky" form as well, the cause of which is seen clearly at higher loads.

At 500gm and 750gm loads a next stage in the deformation process occurs where there is a significant change in the morphology of the groove and material debris. At the base of the grooves there
are scale-like cracks which step-up in the direction of scratch motion (Figure 6.51b,c). These are typically found in surfaces which have been ground by a non-stiff machine, and are known as "chatter" marks. Their formation is due to a stick-slip motion in the grinding process, which usually results in machine vibration. During scratching a high pitched scratch noise was heard. Measurement of the chatter mark separation for the 500gm to 2000gm load scratches gives an estimated audio frequency of around 2.2 kHz to 4.5 kHz, and this is consistent with the noise heard. Larger material fragments of 10μm-70μm size are present in regular intervals along the length of the 500gm load groove (Figure 6.50a). This, together with the fragment structure being in a highly deformed compacted platelet form, suggests that material debris is built up under the diamond tip until it is cast aside. The same characteristics apply to the 750gm load scratch, although the material fragments are larger up to 200μm size due to a larger material displacement and groove site (Figure 6.52). Also, depressions in the groove accompanied by a crack across the groove, suggests a weakness under the groove which caused it to collapse (Figure 6.52a). This is seen more clearly at higher loads.

At 1000gm load a further change in the deformation process occurs. Chevron cracks are randomly formed along both sides of the groove oriented in the direction of scratch motion (Figures 6.55a,b), and the material fragmentation and removal process is enhanced by small and large scale chipping (Figure 6.56a,b). The chevron cracks illustrate the effect of bringing a quasi-static indentation process into motion, where the dynamics of the deformation/fracture process are changed and the radial cracks are distorted. However, the mode of crack propagation remains intergranular (Figure 6.55c). There is a hint of evidence in Figure 6.55b that chatter marks may be linked to the initiation of chevron cracks. From 1000gm to 2000gm the crack lengths range from around 5μm-66μm, increasing to 13μm-99μm, and the angle subtended to all the grooves falls in the same range of around 30°-90°. The large scatter in crack lengths and angles derives from a scratching process that contains a significant contribution from vibrations due to the non-stiff machine. The longest cracks, however, tend to lie at low angles to the groove in a range of around 20°-30°. Material removal due to brittle
fracture occurs via chipping. At the 1000gm load chipping is present as single sites along the edge of the groove and also as "large chip" double symmetrical sites (Figure 6.56a, 6.57a). They are all inclined at low angles to the groove, ranging from $20^\circ$-$50^\circ$, and this is consistent with the presence of long, low angle chevron cracks. Large chips occur where a critical tensile stress results in the propagation and extension of the chevron cracks, causing brittle fracture and material removal. This is determined by the dynamics of the scratch process, principally the indenter size and shape, load applied, speed of traverse, machine stiffness, local surface stress field and the local flaw population. Furthermore, some chip fracture areas have an irregular or "double-bowl" surface which is the result of a complex and variable sub-surface fracture process in the link up of propagating chevron cracks and lateral cracks. Depressions in the groove, first seen at a 750gm load where the depression is only accompanied by a crack lying across the groove, are present again at the 1000gm load (Figure 6.56a,b, 6.57a,b). At this higher load and higher fracture energy the link up of the chevron and lateral cracks creates a fracture path which runs below the groove depth. As the brittle fracture causes the removal of material all the sub-surface detail is clearly seen. To support this evidence, no depressions were found to exist at depths below the chip fracture surfaces. Therefore after the two chip fracture areas were lifted out, the groove bottom collapsed causing a crack across the groove width (the best example of this is seen in Figures 6.59a,b). The material debris, still in a highly deformed compacted structure (Figure 6.56c), mostly lies at a distance from the groove site, suggesting a high energy splintering mechanism of material removal, and the general level of "powder" debris is higher as well. Fragments present in chip fracture areas are probably parts of those areas that have not been splintered away to a distance (Figures 6.56c, 6.57b).

At higher loads of 1500gm and 2000gm, the amount of material debris increases, and the chip sizes are larger and deeper, but are still inclined at small angles to the groove. Also, due to the higher fracture energies nearly all the chip sites are of the double symmetrical type. At 1500gm "plates" of material are still left in the fracture site (Figure 6.58b,c), but at 2000gm the higher fracture energies involved cause most chip fragments
to be splintered away. Other highly deformed compacted material debris is also splintered away at a distance. At 2000gm the deformation-fracture is so concentrated that some new fracture events occur as part of a preceding chip site (Figure 6.60a).

**Diamond Indentor**

The diamond tip was rounded and worn, with damage in the form of cracking and chipping (Figure 6.47, 6.61a). The cracking is a result of tangential and frictional stresses probably causing cleaving of <111> planes during previous scratch tests. The wear of diamond tips in single and multi point grinding is a well known problem (Busch and Prins 1972, Kirchner 1984). This helps to show the approach in the manufacture of diamond grinding wheels, where a resin bond allows old, blunt diamond particles to fall out, thereby exposing fresh sharper diamond particles underneath. After the 25gm - 1000gm load scratching tests had taken place, material debris in powder, small particle, flake and comminuted, compacted form covered the diamond tip, adhering to the surface via attractive electrostatic forces (Figures 6.61b,c 6.62a,b). The diamond tip was also rounded due to the adhesion of highly deformed, compacted material debris (Figures 6.47, 6.62c). Further loading to 1500gm and 2000gm caused a deterioration in the state of the diamond tip (Figure 6.62c).

**Sub-Surface Median Cracks**

Extensive manipulation of the penumbra lighting conditions and photographic processing proved successful in recording the nature of sub-surface median cracks in normal cross-section. The cracks are similar to those found under the 0.4µm CLA ground surface. Underneath a scratch site a semi-circular plastically deformed zone has either one or two cracks which initiate at the plastic/elastic boundary (Figures 6.63, 6.64 and 6.65). The residual stress produced in a ground surface is due to the accumulation of multi particle contact events. Diamond particles at the upper end of the mean size range, or protruding diamond particles will cause enhanced deformation fracture as seen under single point scratches.
7.4 **FURTHER WORK**

The object of the project was to study the deformation/fracture caused by peripheral wheel diamond machining and relate this to fracture strength behaviour. Fine machining should remove all damage from previous machining stages, and it has been shown that this has not been done in the specimens supplied for this project. The act of fine machining inevitably implements machining damage itself and this should be studied in detail without it being confused with previous damage. Structural engineering components require fine machining and an understanding of the nature of machining damage will lead to improved cold strengths. This may be achieved by selective surface treatments such as thermal annealing or ion bombardment which are known to have restored mechanical strengths of components machined to 0.1μm CLA finish (Quinn and Syers 1989).
CHAPTER 8. CONCLUSIONS

A. Processing Flaws
Processing flaws existent in 5wt% Y₂O₃ sinter hipped silicon nitride were examined from two batches of material manufactured at Turner and Newall Ltd and hipped at ASEA Cerama AB (August 1987 and April 1988). Methods of observation included reflection and transmission optical microscopy, scanning and back scattered electron microscopy and energy dispersive X-ray analysis. The following features were identified.

• A three dimensional cellular network is present in billets from both batches of material. Average cell sizes are measured to be 400μm in the 1987 batch and 2.1mm in the 1988 batch. The difference in size is attributed to differences in processing conditions. The formation of the network is a result of flocculation clustering during processing where weak Vander Waal forces cause dominant cluster sizes to form in the colloidal system. The network probably constitutes an area of weakness in the material and evidence of this is seen in the pull-out caused in these areas on mechanically polishing the surface.

• Clusters of 1 - 3μm metallic particles are present in both batches of material. In the August 1987 batch the cluster size ranges from 5 - 45μm. The contaminant particles are steel and were introduced as a result of the ball milling process which employed a steel ball mill. In the April 1988 batch the cluster size ranges from 3 - 25μm.

• Glass encapsulation failure during hipping caused extensive surface porosity at one end of an August 1987 HIP billet.

B. Machining Damage
The macro and microscopic features and effects of machining damage caused by peripheral wheel diamond grinding flexural rupture test bars to three surface finished were studied. Material from the April 1988 HIP batch was used and methods of observation included: normal and penumbra illumination reflection optical microscopy, transmission optical microscopy, scanning, backscattered and transmission electron microscopy. A
profilometer was used to measure surface roughness.

Coarse Machined Surface

- Deep grooves up to 2μm depth, 18μm width are superimposed on the general 0.35μm centre line average surface roughness by singularly large diamonds in the 350 grit diamond wheel.

- Sub-surface median cracks normal to the machining direction were identified from cross-sections from one machined bar using penumbra illumination optical microscopy, an undocumented technique. Semi-elliptical in shape they extend from 6μm to 45μm depth below the machined surface and range from 19μm to 101μm in length parallel to the machining direction. The average crack site separation normal to the machining direction is 227μm and parallel, 413μm. An estimation of the area surface crack concentration is therefore approximately 18cm⁻².

- The sub-surface median cracks initiate at the focal point of a tributary system of micro cracks at an average depth of 4 - 5 μm below the machined surface. The tributary system is 15 - 16μm wide at the machined surface. It is believed that the median cracks initiate at the plastic/elastic boundary of the plastically deformed surface layer.

- Therefore the residual compressive layer, formed by the overlap of localised residual stresses from multi-particle contact events and bound by an underlying tensile field has an average depth of 4 - 5μm.

- TEM microdiffraction analysis of normal cross-sections reveals that Bragg diffraction spots are distorted by the high lattice strains and mechanical damage at the machined surface. Measurements of the extent of arcing or streaking provides an estimation of the dislocation density, found to be of the order of 4 x 10¹⁴ m⁻². This effect tends to disappear at a depth of 4 - 5μm below the machined surface and this is further evidence of the existence of a layer in residual compression with an average depth of 4 - 5μm.
Fine Machined Surface

- Fine machining with a 600 grit diamond wheel produced a surface of 0.01/0.02μm CLA roughness. However, evidence of machining damage is still present in the form of "remnant tracks" which have a length of up to 1.5mm, and widths of 16μm which is consistent with the width of the tributary system of microcracks and the deep groove widths in the coarse machined surface of 18μm. The tracks lie parallel to the machining direction and consist of material pull-out. They are remnants of machining damage under grinding grooves introduced in previous machining stages.

- Both the intermediate and fine machined bars show a convex surface curvature across the width of the bars. An approximated radius of curvature for each surface finish was found to be 0.9m to 1.2m, and 9m to 12.6m respectively. It is suggested that this is due to the effect of the residual surface compressive stress.

Modulus of Rupture Tests and X-ray Microdiffraction Tests.

- Work by Quinn (1989) shows that an increase in the quality of surface finish is accompanied by an increase in the mean strength and Weibull modulus.

- A distinct anisotropy in the fracture strengths parallel and normal to the machining direction for the coarse machined surface is evidence of anisotropy in machining damage formed by a peripheral diamond grinding wheel.

- Anisotropy in fracture strengths from the fine machined bars shows that machining damage from previous machining stages was not completely removed.

- X-ray microdiffraction tests by Flewitt (1989) show that machining damage produces a long range biaxial residual compressive field with the highest component acting normal to the machining direction.

Polishing a Coarse Machined Surface

To illuminate the effects in producing a fine machined surface from an initial coarse machined surface, a coarse machined bar
was polished, removing material layer by layer. The effect of polishing a surface containing an indentation was also observed.

- Once the coarse machined surface roughness and deep grooves are removed by fine polishing, finite length remnant material pull-out tracks similar to those present in the fine machined surface are formed. The effect of mechanically polishing an area of deformation is also seen in the removal of an indentation. A "band" of material pull-out is formed around the indentation site.

**Single Point scratch Tests**

To help understand the damage mechanisms in multi-point diamond wheel machining the effects of implementing a series of single point Vickers pyramid diamond scratches in a polished surface was studied.

- From low load (25gm) to high load (2000gm) the single point scratch tests create an increasing level of surface damage. This changes from grain boundary fracture and sub-micron particle debris to large fragments of compacted material up to 200µm in size and large chips.

- At loads of 500gm and above scale-like cracks or "chatter marks" are formed at the groove bottom which step up in the direction of scratch motion. The stick-slip motion produces crack separations around 2.5µm which approximate to audio frequencies of around 2 to 4.5 kHz, consistent with a high pitch scratching noise heard during the tests.

- At 1000gm loads and above chevron cracks are formed on the surface in the direction of scratch motion. These are precursors to "chipping" where material chips are lifted out of the surface. These fracture sites are accompanied by depressions in the groove bottom which have a crack lying across the groove width. It is suggested that the groove bottom collapses after the creation of a chip site. The length of chevron cracks and size of chip sites increase with higher loads.

- Cross-sections of the scratched surface show that the
deformation/fracture is similar to that caused by singularly large diamonds in the coarse machined surface. However here the median cracks that are present under grooves initiate beneath a roughly semi-circular zone of deformed material at the plastic elastic boundary.

- As a result of scratching the diamond tip became round and worn, with damage in the form of cracking and chipping. It was also covered by compacted, comminuted Si$_3$N$_4$ material.
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A.1 REACTION BONDED SILICON NITRIDE (RBSN)

Fabrication

Turner and Newall Technology Ltd. produce reaction bonded silicon nitride (NITRASIL) by nitriding a silicon powder compact in molecular nitrogen at about 1400°C. The spray dried metallic silicon powder is carefully chosen to be 5 - 40 μm in size as the particle size affects the nitridation reaction and the finished material's mechanical properties. A small particle size results in a fine grained material, but too fine a powder makes the compact difficult to nitride. Also, if the powder is too pure the nitridation reaction does not occur, and this is solved by introducing sintering additives comprising 1 - 3% Fe, 0.2% Ca and 0.5% Al. To help compaction a PVA binder is also added at this stage. The initial forming of the material is carried out by filling a rubber mould and cold isostatic pressing (CIP) with hydraulic fluid in a pressure vessel, to 105 - 175 MPa over several minutes. The material now takes the form of a "fragile" compressed powder held together by the binder, and is in the "green" stage condition. The binder is now burnt out in a gentle process at a temperature of 500°C over several hours. Injection moulded components contain up to 30% binder, and this is very difficult to remove completely without structural damage. The component is now weaker and requires presinter treatment, where soaking is carried out in Argon at 1100°C. The green condition strength is returned without any shrinkage. At this stage components can be carefully handled, stored or green machined to shape prior to nitriding. The final stage of production takes place in the nitriding unit, with the reaction commencing as the temperature reaches 1400°C. The reaction is exothermic which
initially causes a high demand for nitrogen. The temperature must be carefully controlled though as melting of the silicon powder compact may result due to over heating, thus rendering impossible a complete conversion of the powder. The reaction can be monitored by weight gain and complete conversion should theoretically be 66.5%. Another monitor is the nitrogen demand of the reaction. The complete process takes 5 - 7 days.

Nitridation causes minimal shrinkage of the green stage component as the crystals of silicon nitride tend to fill the porosity of the compact. However, as the penetration of the gas to the centre of the component must be maintained throughout the conversion process, the material usually has an open porosity of 18 - 25%. The density ranges approximately from 75-80% of the theoretical density, 3.19 gm cm$^{-3}$.

RBSN components are simple and cheap to fabricate as extensive final machining is rarely needed due to the low shrinkage and maintenance of the original green stage component dimensions.

The microstructure consists of submicron crystals of $\alpha$ and mostly $\beta$ silicon nitride. The $\beta$ phase exists as elongated prismatic grains which are hexagonal in cross-section. The largest grains can reach 5 - 10$\mu$m in length and 1 - 2$\mu$m in diameter. The random orientation of the grains produces a strong interlocking type structure which is enhanced by a high aspect ratio. There is considerable interconnected porosity, where the larger pores (up to 50$\mu$m in size) tend to result from voids in the original compact or from melting of silicon particles during nitridation. These are interconnected by channels which are in the size range from 0.01 - 1.0$\mu$m in diameter. A whisker morphology of $\alpha$-Si$_3$N$_4$ usually develops at external surfaces rendering the as-nitrided material a pale grey in colour, whereas the interior is generally dark grey.

Applications
RBSN is widely used in light metal foundries, its hardness, thermal shock resistance and non-wetting properties make it suitable for modern metal pumps, pipes, valves, pouring spouts, crucibles and continuous casting dies, and its reliability increases the scope for automated molten metal handling. Its
thermal shock resistance, dimensional stability and electrical insulation allow jigs and fixtures to be made for use in induction heating and vacuum brazing. RBSN is also used in instrumentation and inertial navigation equipment, where its low coefficient of expansion ensures exact calibration through a specified temperature range. In chemical plants its stability is beneficial for use as seals, ladles, spray deflectors and process equipment.

There is current development for applications in pistons, pre-combustion chambers, turbine blades and exhaust diffusers. These would replace complex components containing strategic metals and operate at much higher temperatures. Research is also being carried out on radomes, disc brakes and on compounds for use in rockets and military weapons (Figure A.1a).

A.2 SINTERED REACTION BONDED SILICON NITRIDE (SRBSN)

Fabrication

The initial reaction bonding process is exactly the same as for RBSN, except that sintering additives $Y_2O_3$, $Al_2O_3$ or $Cr_2O_3$ are added to the initial powder prior to CIP. The RBSN component still has 20% open porosity after nitriding, but heat treating at a temperature of 1100 - 1800°C reduces this to microporosity. The sintering process is maintained at this temperature under a nitrogen gas pressure of a few psi above atmospheric pressure for around five hours.

The finished component undergoes approximately 11% shrinkage, and this has to be compensated for by green machining over size, but final machining is usually required.

The microstructure consists of fine $\beta-Si_3N_4$ grains around 1µm in diameter with the largest reaching 10µm. The intergranular phase can be either amorphous glass or crystalline, depending on the composition of the sintering additives and the reaction process conditions.

Applications

SRBSN has applications in a wide range of engineering applications similar to RBSN.
Figure A.1a  Silicon nitride missile cones

Figure A.1b  Silicon nitride rotors
A.3 SINTERED SILICON NITRIDE (SSN)

Fabrication
The fabrication of SSN is quite different from that of RBSN and SRBSN in that the starting powder is silicon nitride plus MgO, Al₂O₃ or Y₂O₃ sintering additives, and no nitridation process is required.

At Turner and Newall Ltd. sintering is carried out at a temperature of 1700 - 1800°C, producing a material similar to SRBSN, although with 20% shrinkage. However, more complex shaped components can be fabricated with SSN using CIP, injection moulding or slip casting forming techniques.

Applications
The performance of dense, hard, strong lightweight SSN is superior to that of RBSN. It is more resistant to thermal shock, a better electrical insulator and has strength retention up to 1000°C. It is used as a bearing material at high and low temperatures, and as metal forming dies and cutting tools. It has good wear resistance against cast iron in dry and lubricated corrosive and erosive environments.

Further applications include brazing, soldering and welding fixtures, MIG, TIG and plasma welding nozzles, electrical insulation components, metrological jigs and fixtures. Developments are currently being carried out to replace metal components in gas turbines (ie rotors, Figure A.1b), and reciprocating engines (ie valve trains and rocker-arm pads). Current uses are in diesel engine components such as pre-combustion chambers and glow plugs.

A.4 HOT PRESSED SILICON NITRIDE (HPSN)

Fabrication
The fabrication of hot pressed silicon nitride is similar to that of hot isostatically pressed silicon nitride, (See Section 2.3). An α silicon nitride powder with Y₂O₃ or MgO densifying additives is compacted and sintered at a temperature of 1800°C under a uniaxial pressure of 30 MPa. At Norton Company, NC 132 HPSN contains 1 - 5 Wt% MgO depending on the requirement for refractoriness. On cooling a Mg-Si-O-N intergranular glassy phase is formed. The β-Si₃N₄ grains range from 0.5 -
2\,\mu m in diameter and are 1 -5\,\mu m long, and a few reach 10\,\mu m length. The uniaxial pressure produces a smaller effective grain size parallel to the pressing direction.

**Applications**

HPSN is a higher performance material than SSN and is used in several engineering components for the processing industry. Examples are bearings, seals and valves and high temperature engineering components. Other HPSN materials are also used in ball bearings for abrasive or corrosive environments and in rotary valves for piston engines and dies for resistance sintering.

**A.5 SIALON**

**Fabrication**

Aluminium can replace silicon in the beta silicon nitride structure if, at the same time, nitrogen is replaced by oxygen giving a substituted solid silicon alumina oxynitride solution referred to as $\beta'$-sialon, with a range of composition:

$$\text{Si}_{6-z}\text{Al}_2\text{O}_{2-z}\text{N}_{8-z}$$

based on the $\beta$-$\text{Si}_6\text{N}_8$ unit cell.

Sialons are synthesised by reacting together silicon nitride with other constituents to achieve an appropriate $\beta'$ composition. Yttrium oxide is used as a liquid phase sintering aid and, while it should be possible to produce single phase $\beta'$-sialon, densification considerations require that the material's chemistry is such that the final assemblage is multi-phase. A silicon yttria alumina oxynitride composition is produced. Two major material types have been developed commercially by Lucas-Cookson Syalon, one consisting of $\beta'$ grains with a residual glass, the other consisting of $\beta'$ grains and a semi-continuous intergranular crystalline phase of yttrium aluminium garnet (YAG).

**Applications**

Syalon 101 and 201 are Lucas Cookson Syalon products. Syalon 101 is used in many engineering applications where operating temperatures do not continuously exceed 1000°C. Typical
applications are extrusion tooling, drawing dies and plugs, welding components and industrial wear parts. Syalon 201 can operate in environments where the operation temperature extends to 1400°C. Examples are gas turbines (blades, rotors, shrouds, combustors), diesel engines and molten metal handling situations.

Up to now one of the most successful applications has been as a cutting tool for machining metals. The performances of Syalon tool tips in cutting cast iron, hardened steel and a nickel-based alloy are compared with those of cobalt-bonded tungsten carbide and of alumina in Table A.1.

Table A.1: CUTTING PERFORMANCES OF COBALT-BONDED TUNGSTEN CARBIDE, ALUMINA, AND SYALON.

<table>
<thead>
<tr>
<th>WC</th>
<th>Cast Iron</th>
<th>Hardened Incoloy Steel EN31 901</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cutting speed, ft/min</td>
<td>800</td>
</tr>
<tr>
<td></td>
<td>Depth of cut, in</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>Feed rate, in/rev</td>
<td>0.02</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>Cutting speed, ft/min</td>
<td>2000</td>
</tr>
<tr>
<td></td>
<td>Depth of cut, in</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>Feed rate, in/rev</td>
<td>0.01</td>
</tr>
<tr>
<td>SYALON</td>
<td>Cutting speed, ft/min</td>
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</tr>
<tr>
<td></td>
<td>Depth of cut, in</td>
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</tr>
<tr>
<td></td>
<td>Feed rate, in/rev</td>
<td>0.02</td>
</tr>
</tbody>
</table>

The "lead time" for machining some of the RB211 aeroengine turbine discs is said by Rolls Royce to have been reduced to less than one-quarter by using Syalon inserts. Other products include welding components and location pins for the resistance welding of captive nuts on vehicle chassis. The usual hardened steel pins in alumina insulating sleeves have lasted for 7000 operations (ie a shift) while Syalon pins have completed 5 million operations (one year) without signs of wear.
With Syalon sintered to almost the final required dimensions, seals and bearings are produced which are wear resistant, hard and have good tribological properties. Syalon die inserts used in the extrusion of brass, copper, bronze, aluminium, titanium and steel give good surface finishes, dimensional accuracy and high extrusion speeds. Syalon copes with a wide range of wear environments in contact with metals with or without lubrication and tube-drawing die and mandrel plugs are examples where there are marked increases in productivity compared with the conventional use of tungsten carbide. As Syalon is resistant to most molten metals including steel (although attacked by slag) it is used in casting and metal spraying. Slip-cast Syalon coracles are used in pulling single crystals from a gallium phosphide melt which attacks most other materials.
APPENDIX B
SPECIMEN PREPARATION

CONTENTS
B.1 Sampling
B.2 The Struers Accutom Cutting Machine
B.3 Talystep Stylus Instrument
B.4 Definition and Estimation of Centre Line Average Roughness Parameters
B.5 Grinding and Polishing
B.6 Disc Coring
B.7 The VCR Dimpler and Mechanical Pre-Thinning

B.1 SAMPLING
B.1.1 Introduction
To examine silicon nitride ceramic components they are often cut into a manageable size and shape. It is important that minimum deformation of the material occurs during this process, so that the cut surface can be directly ground and polished, suitable for examination. The most widely recognized sampling method uses a diamond cut-off wheel. This method is relatively fast and efficient, considering the hardness and also brittleness of these materials. Cutting-off is a mechanical cutting method in which a motor-powered abrasive wheel cuts through the material in a liquid. Wet cutting should eliminate dust problems and suppress heating of the cut surface.

The cut-off wheels consist of a bond, either metal or bakelite, in which diamonds are dispersed. The cutting properties of the wheel are determined by the size, shape and distribution of the diamond grains, while the wearability depends on the bond. For hard ceramics a "soft" wheel is used, where the worn grains break out during cutting, and the binder wears down rapidly thus exposing new sharp grains.
B.1.2 Quality of Cut Slices with (i) Rotating Specimen Motion (ii) Stationary Specimen

(i) An NC 132 HPSN flexural rupture test bar of dimensions 3mm x 3mm cross-section and 50 mm length was cut into twenty pre-set 0.5mm slices with a Struers Accutom cut-off machine (see B.2) with the specimen set in rotational motion. A high concentration, continuous rim, steel bond 350 grit diamond cut-off wheel of 125mm diameter and 0.7mm thickness was used with a wheel speed of 1000 r.p.m., a feed rate of 0.2mm/min and a constant drip of an alcohol-based lubricant. For each new cut the micrometer was set forward 1.2mm to cut off a 0.5mm slice. The standard deviation of thickness was only ± 14 microns, showing that the wheel vibration is low and a specimen slice can be cut reproducibly. However, there is a major drawback in this method of sampling. A spigot is left protruding from the cut surface on the axis of the specimen rotation. Its formation occurred as the wheel approached the remaining material at the centre of rotation. Even the pressure due to a slow feed rate of 0.1mm/min was enough to snap the cut specimen from this last attachment. This may have caused large stresses in that area of the specimen and further damage may be caused when the spigots are removed by grinding. Unless thick slices are produced (which is impractical for TEM preparation), the formation of spigots is an unacceptable risk in the production of damage-free specimens.

(ii) An NC132 HPSN bend test bar of 3mm x 3mm cross-section was bonded on a bakelite mount with an araldite epoxy adhesive and left to cure for 24 hours. This was cut directly, with no specimen rotation, with a metal bond, 75mm diameter, 150μm thick diamond cut-off wheel. A slow feed-rate of 0.1 - 0.25mm/min and a wheel speed of 1000 r.p.m. was used, and as the specimen was kept stationary a fast lubricant drip rate was positioned over the wheel/material surface contact point to try and remove any build up of material debris. Thirteen slices, each pre-set with the machine micrometer, were cut in a period of approximately three hours.

A burr was left on most slices at the point at which the wheel
was just about to cut through cleanly. The distribution of thicknesses is higher than for (i) with $\sigma = \pm 34$ microns, and is probably due to greater wheel vibration due to the entrapment of material debris at the cutting edge. The glue bonding the bar to the bakelite holder was found to be rubber-like in texture, and this explains the formation of the burrs. A harder glue would have strongly secured the slice while the final micrometers of material were cut through. However, the drive of the wheel caused the slice to splinter off, as in Section i) where spigots were formed with a rotating specimen. Another feature of the stationary specimen technique are the curved surface cut lines which follow the circumference of the wheel cutting edge and cover all the specimen surface. Their presence may indicate deep underlying sub-surface damage. Thus the general surface roughness and step heights were analyzed with a Talystep stylus instrument (see B.3) to gain a "feel" as to how much material should be removed to achieve a damage and stress-free surface.

The surface profile of the cut face was recorded with the stylus traversing normal and parallel to the cut lines (Figure B.1a, B.1b), giving the following roughness parameters:

**Normal:** Maximum Peak to Valley Height, $R_{\text{max}} = 2.5$ micron  
Centre line Average Roughness, $R_a = \pm 0.30$ micron

**Parallel:**  
$R_{\text{max}} = 0.6$ microns  
$R_a = \pm 0.09$ microns

It can be seen from Figure (B.1a) that the lines are unsymmetrical peaks with a gradual rise followed by a steep, straight decline. This type of surface is created by a material "cut and break" mechanism which is dependent on many factors, but principally the diamond wheel feed rate. The profile parallel to the cuts (Figure B.1b) is not as rough as the normal profile, and so it is clear that the majority of deformation occurs tangential to the cutting wheel and is mainly due to the wheel feed rate. The maximum peak to valley height of the "saw-tooth" peaks is 2.5 microns and is small compared to the average slice...
Figure B.1a

Figure B.1b

Figure B.1  Profile of an as-cut surface with the stylus pick-up traversing a) normal and b) parallel to cut lines
thickness of 495 microns. The cut surface can then be mechanically removed by diamond grinding to produce a flat surface (See Section B.5).

B.2 THE STRUERS ACCUTOM CUTTING MACHINE
The Accutom is a small scale laboratory precision cut-off machine Figure B.2a). The cutting table is hydraulically driven and automatically feeds the specimen towards the cutting wheel with a continuously variable speed from 0.1 to 5mm per minute. The speed of the cutting wheel is continuously variable from 100 to 1000 r.p.m. This to can be adjusted so that a rapid cut is attained without distortion of the wheel, producing an undamaged material surface.

The maximum length of cut is 75mm, corresponding to the travel of the table, and a micrometer screw gauge of range 0.01 to 25mm allows precision cuts to be made. The specimen is secured in a clamp which rotates, and with a constant supply of lubricant a fresh and clean cutting surface is ensured; this avoids the possible build up of material debris at the wheel/material surface contact point and also on the wheel.

B.3 TALYSTEP STYLUS INSTRUMENT
The Talystep (Figure B.2b) was designed for use in the microelectronics industry for thickness measurements, in metallurgical research and micro-topographical analysis. Surface roughness is revealed when the stylus is traversed across the specimen surface. The vertical movement of the stylus is amplified electronically and recorded graphically.

The specimen is placed in the centre of a work stage under the viewing microscope of magnification 10 x. Two micrometers move the work stage in two directions at right angles for precise positioning of the specimen relative to the pick-up stylus. A pre-tension is set by adjusting the stylus force control which controls the force with which the stylus bears on the specimen surface. This is done with the pen shift set at zero. The pen shift adjusts the position of the graph pen to enable the graph to be recorded in the required position across the width of the chart. The pick-up is lowered until the pointer of the meter, which indicates the stylus force, moves to mid-scale. There are
Figure B.2a Struers Accutom Cutting Machine

Figure B.2b Talystep Stylus Instrument
eight magnifications of the vertical movement of the stylus: $5 \times 10^3$, $1 \times 10^4$, $2 \times 10^4$, $5 \times 10^4$, $1 \times 10^5$, $2 \times 10^5$, $5 \times 10^5$ and $1 \times 10^6$, and three traverse speeds, over a maximum distance of 2mm, giving horizontal magnifications on the rectilinear recorder of 50 x, 200 x and 2000 x. The stylus force is set according to the vertical magnification to be used. By setting a vertical magnification of $5 \times 10^3$ and a horizontal magnification of 50 x the specimen is tested to ensure the surface is set parallel to the line of pick-up traverse. If the specimen surface is inclined relative to the plane of the pick-up stylus movement, the graph will be inclined relative to the chart scale and will be difficult to assess accurately. Adjustments are made with the work stage tilt controls via abutment screws and a refined, step by step levelling procedure with increasing vertical magnification is carried out. There are three waviness filters which suppress the higher frequencies of pick-up output, representative of residual roughness, so that general levels of the upper and lower surfaces of the specimen can be more readily assessed. There is also one roughness filter which suppresses the lower frequencies of the pick-up output to enable the surface roughness of the specimen to be assessed. With a traverse dial indicator of 0.02 mm divisions an area of the specimen can be selected for measurement. A low vertical magnification is first used and then with increasing magnification an optimum profile is attained on the recorder chart.

At the higher magnifications the minimum stylus force may be limited by floor vibration and acoustic noise, both of which tend to bounce the stylus off the surface resulting in spurious kicks on the graph. Usually, a conical 0.0125mm radius stylus tip is used for step height measurements but for surface roughness measurements a specially sharp stylus is employed. The most suitable form of stylus for ground and lapped surfaces having uni-directional lay has a truncated pyramidal tip of approximately 0.1µm x 2.5µm maximum value, mounted so that the 0.1µm radius is presented in the direction of traverse.
B.4 DEFINITION AND ESTIMATION OF CENTRE LINE AVERAGE ROUGHNESS PARAMETERS

The roughness parameters are defined as follows and are illustrated in Figure B.3.

The maximum peak to valley height $R_{\text{max}}$ is the largest single peak to valley height.

The centre line average roughness $R_a = \frac{1}{L} \int_{0}^{L} |z| \, dx$

where $L = \text{sample length}$.

To estimate the centre line average roughness a line is drawn approximately through the centre of the profile, so that roughly half of the peaks are above and half below. This does not need to be exact as the integral expression in the equation sums over an area. Thus $\int_{0}^{L} |z|/dx$ is the area under the profile to the centre line. If a 1.5mm sample length is to be analysed for example (Figure 6.1a), it is divided into sixty $dx = 25\mu m$ strips. If the Z axis is divided into 1$\mu m$ segments then each small division is equivalent to 0.2$\mu m$. The area of each box is $25\mu m \times 0.2\mu m$ and the total number of boxes are calculated for each strip $dx_1$, $dx_2$, ..., $dx_n$. Finally the total area under the profile is summed up and divided by the sample length $L$ to give an estimation of the centre line average roughness. The process is laborious and time consuming but provides a reasonably accurate calculation of surface roughness.

B.5 GRINDING AND POLISHING

B.5.1 Introduction

For surface examination by reflection optical microscopy and scanning electron microscopy, or the preparation of a petrographic thin section for transmission optical microscopy, the Accutom cut-off machine leaves a surface quality which obviates the need for a "fine grinding" stage before the polishing process (fine grinding is taken as the grinding required when the centre line average roughness is below 1$\mu m$). A polishing process could immediately be employed but would be too time consuming. Not only must the surface deformation be removed to produce a flat surface, a depth of material which has
Figure B.3  Diagram of roughness parameters
undergone sub-surface damage must also be removed. A fine grinding stage is both fast and efficient.

A free abrasive on a hard lap, such as silicon carbide powder of grain size 10 -20μm on a cast iron lap, has been used. However, the free motion of abrasives cause too much pull-out and sub-surface damage. Silicon carbide wet paper of the same grain size range also damages the surface and sub-surface and the removal rate is slow.

For such a hard and brittle material the best known method of surface wear is by grinding on a soft lap in which the abrasive is partially embedded in the surface. A diamond spray on a synthetic disc such as the Struers Petrodisc minimises pull-out while maintaining a high removal rate and good edge retention. Circumferential grooves and an oil-based lubricant ensure that the material debris is efficiently removed thus avoiding further pull-out.

A diamond abrasive on a cloth lap has been used for the polishing stages, but a flat surface is not produced due to the cloth being compressible. The cloth tends to conform to the surface of the specimen under the slightest pressure. The primary and secondary phases of the material differ greatly in hardness and as the abrasive is kept in contact with all parts of the surface, the softer phase is removed at a faster rate during the long polishing times. This relief polishing causes the loss of sharpness of edges and the grains become rounded. A soft lap with a diamond abrasive partially embedded in the surface is again a good method. For silicon nitride a grooved, commercially pure tin lap (99% pure) has been very successful in producing a flat surface free from surface relief at a fast polishing rate. Work by Smith (1982) has shown that a spiral groove machined on a fine turned tin lap optimises the performance. The groove spacing was 1 - 2mm with a depth of 0.55mm and an included angle of 60°. Work at the University of Surrey has been carried out using a tin lap with the same dimensions but with circumferential grooves as this was thought to be adequate. However, all grinding and polishing techniques cause a certain amount of pull-out of grains and groups of grains. In order to minimise the pull-out it is necessary to ensure that the damage introduced in
each polishing stage is removed completely by the next polishing stage. The difference between porosity and grain pull-out can be difficult to distinguish though. When the material surface is viewed under a microscope the porosity appears as ovular or circular-shaped voids. Pull-out voids have generally the same dimensions but are irregular with sharp edges. The void shapes that are between the two are difficult to distinguish. The only test for successful minimisation of pull-out is repeated microscopic examination of the total apparent porosity. Polishing must continue until no further decrease in apparent porosity occurs.

A polishing sequence of 6μm, 1μm and 0.25μm produces a flat surface with minimum pull-out and without surface relief. However, scratches which are formed on the material surface cannot all be removed, even with a 0.1μm diamond compound. Polishing with a 1μm diamond compound on a soft cloth or polishing on a Dimpler machine with a soft polishing pad has been successfully used in removing the surface scratches.

B.5.2 Grinding
a) NC132 HPSN specimens cut with the Accutom cut-off machine (See B.1.2) were mounted evenly on four bakerlite holders with cyanoacrylate adhesive and ground for a few seconds by hand on 220 grit silicon carbide paper to remove the burrs. Grinding was then carried out with a Petrodisc on a Struers Planopol/Pedamax 2 automatic grinding machine, which gives good flexibility in the control of the grinding speed, force and length of time. The mounted specimens were clamped in four corners of a carousel which fits on the axel of the machine. The 290mm diameter Petrodisc was sprayed with 14μm diamond compound, lubricated with an oil-based liquid and set to rotate at 150 r.p.m. An applied total pressure of 120 N on the 3mm x 3mm cross-sections produced perfectly flat surfaces. However, the high specimen loading of 0.89 Nmm⁻² caused extensive chipping on many surfaces. Further grinding under a total applied load of 30 N (specimen loading of 0.22 Nmm⁻²) gave a material removal rate of 4 μm/min and produced a flat, undamaged surface. Repeated experiments gave a removal rate of 4 - 4.5 μm/min.

b) A 5 wt% Y₂O₃ additive sinter hipped silicon nitride billet of
dimensions 21.1 mm diameter by 5 cm length was cut into slices with an Accutom cut-off machine. Each cross-section was mounted with cyanoacrylate adhesive on a bakelite holder and ground by hand on Struers Petrodisc rotating at 150 r.p.m. using an oil based lubricant. The lower specimen loading gave material removal rates of 2.0 - 2.5 \mu m/min, although occasionally rates around 1.3 and 3.1 \mu m/min were produced. This was due to variations in the grinding conditions; concentration of diamond compound, hand pressure applied to the specimen and volume of lubricant used. A typical surface (Figure B.4), is similar to the one produced in a) although there are less scratch marks due to a lower specimen loading.

The surface profiles of the ground surfaces were recorded on a Talystep stylus instrument at a vertical magnification of 1 \times 10^5 a horizontal magnification of 2000 x (Figure B.5). An analysis of the profiles gave an average estimated centre line surface roughness of 0.07 microns and a maximum peak to valley height of 0.75 microns.

B.5.3 Polishing

B.5.3.1 6 \mu m Diamond Polishing on a Tin Lap

The as-ground specimen cross-sections from B.5.2 b) were mounted on glass slides with cyanoacrylate adhesive, and polished on a grooved tin lap (stationary) with a 6\mu m diamond spray compound and an oil-based lubricant, giving material removal rates of around 1.1 microns/min. Polishing was carried out until all the surface roughness from the previous grinding stage was eliminated and until the total apparent porosity reached a constant level, thus ensuring the pull-out was kept to a minimum (Figure B.6). Surface scratches are present and this is an inevitable artefact from tin lap polishing.

An analysis of the surfaces with a Talystep instrument (Figure B.7) shows that the surface roughness has been considerably reduced from 0.07 micron to 0.026 microns with a maximum peak to valley height of 0.17 microns. In the centre of most of the cross-sections there appeared a different material phase in the form of white speckles. During polishing these white speckled areas suffered greater amounts of surface pull-out than the rest of the specimen surface area (See Section 3.3.1.1 ii).
Figure B.4a  

Figure B.4b  

Figure B.4c  

Figure B.4d  

Figure B.4  SEM micrographs of Petrodisc 14μm diamond ground surface taken at a) 0°, 1,000x  b) 60°, 3,500x  c) 60°, 9,000x  d) 75°, 8000x
Figure B.5 Profile of a Petrodisc 14μm diamond ground surface at vertical magnification of $1 \times 10^5$
Figure B.6 SEM micrographs of tin lap 6μm diamond polished surface taken at a) 0°, 1,000 x b) 60°, 3,500 x c) 60°, 9,000 x d) 75°, 8,000 x
Figure B.7  Profile of tin lap 6μm diamond polished surface at a vertical magnification of 2 x 10^3 x
It was also noted that after polishing a number of specimens a very thin black slurry would form on the surface of the tin lap. The black slurry is due to material debris mixing with the applied diamond compound and lubricant. Most of the material debris is deposited in the lap grooves but a film does build up on the lap surface. It was found that when this happened the amount of pull-out in the specimen surface was greater.

B.5.3.2 1 μm Diamond Polishing on a Tin Lap
The 6 micron-polished cross-sections, mounted on a glass slide, were polished on the grooved tin lap with a 1μm diamond spray compound and an oil-based lubricant. Polishing on a stationary lap gave slow removal rates of lower than 0.01 μm/min. Although a mirror-like surface was produced, on a microscopic scale surface scratches were still present and the total apparent porosity appeared to be higher than expected for a fine 1μm polish on a silicon nitride ceramic containing porosity of less than 1% (Figure B.8). Polishing with a soft cloth eliminates these scratches, but surface relief is inevitable. Analysis with a Talystep instrument shows the surface profile to be very flat (Figure B.9), with the CLA roughness at the region with pores = 11.5nm, and the smooth region = 2nm. As a result of this flatness, atomic number contrast effects due to the different material phases become apparent at a tilt of 0 deg (Figure B.8a).

B.6. DISC CORING
B.6.1 Introduction
Specimens to be used for TEM analysis must be less than 3mm in diameter to fit in the microscope specimen holder. Thus a method of sectioning 3mm diameter discs with minimum material damage is required. Various methods have been used on different materials. Although silicon nitride has no preferred cleaving plane such as gallium arsenide, Mr D Clinton of the National Physical Laboratory and Professor M Lewis of Warwick University have used this technique to produce 3mm diameter discs. A 3mm copper grid is bonded with adhesive on top of a thinned section and the material is cleaved around the grid circumference. However, experience has shown that more often than not the crack travels to the centre of the disc rather than around the edge, therefore making the method time consuming by a low success rate. An etching technique has been used by Puttick and Rudman (1970)
Figure B.8 SEM micrographs of tin lap 1μm diamond polished surface taken at a) 0°, 1,000x  b) 60°, 9,000x
d) 75°, 8,000x
Figure B.9a Profile of tin lap 1µm diamond polished surface at a vertical magnification of $2 \times 10^5$

Figure B.9b Profile at a high vertical magnification of $1 \times 10^6$ showing "local" surface roughness
on cadmium where a thin section has a 3mm diameter protective solvent applied to the surface and the rest of the specimen is dissolved away by an etchant. One major disadvantage in this method for silicon nitride may be the severe effects of etching, where the intergranular phase is primarily attacked. D Clinton of NPL has found that for longer etching times no intergranular phase is left in the surface and sub-surface. A widely used technique in the production of ceramic TEM specimens employs a coring tool. Coring is a mechanical cutting method where a rotating coring tool tip cuts through the material in a diamond slurry. The tool is in the form of a cylinder of 3 mm inside diameter and a wall about 1 mm thick, with slits up the side to allow free movement of the slurry around the cutting point. An applied diamond paste combined with an oil-based lubricant forms the slurry which acts as the abrasive medium. The tool is made of brass so that diamonds become impregnated in the soft metal tip thus providing the cutting action (the same material removal mechanism as for the Petrodisc grinding and soft tin lap polishing (See B.5.1, B.5.2, B.5.3).

B.6.2 Disc Coring with a Drilling Machine
Thin specimens of about 500 to 200 microns were bonded on glass slides with cyanoacrylate adhesive. The adhesive should be spread evenly underneath the specimen as a support is needed when the final micrometres are cut through, thus avoiding the formation of burrs or chipping of the surface. The glass slides were held firmly in a clamp and placed on the centre of the work base of a standard Elliot Progress drilling machine. The centre of the work base has a 3cm hole which allows the progress of the cut to be followed from underneath the work base. Using a tool speed of 2570 r.p.m. with a 25 micron diamond paste and oil-based lubricant, cutting was carried out manually by bringing down the tool head to the specimen surface with the lever. Great care is needed in applying pressure as fractured specimens are a common occurrence as a result of driving down the tool head to accelerate the rate of cutting. It is also beneficial to frequently raise and lower the tool head as the build up of debris prevents the diamond impregnated tool tip from cutting efficiently. As silicon nitride is transparent to light the coring can be carefully controlled just before the final micrometres are cut through. A specimen 500 microns thick should
take approximately 5 minutes to core. If, alternately, a VCR Servo Drill is available, then a higher specimen production success rate is achievable. The VCR Servo Drill was originally designed in Silicon Valley, Ca for use in the drilling of semiconductor materials. It is the vibration free, precision drilling that makes it so useful for the coring of a hard and brittle material such as silicon nitride ceramic.

B.7. THE VCR 500 Dimpler and Mechanical Pre-Thinning

The Dimpler machine (Figure B.10) was originally developed by electron microscopists in Silicon Valley Ca for the TEM specimen preparation by mechanical pre-thinning of hard materials such as silicon and sapphire. Its application has extended to hard, multiphase materials, soft metals, alloys and semiconductors. Pre-thinning with the Dimpler reduces specimen preparation time as a thinned specimen will not require lengthy ion beam milling.

The specimen is mounted on the specimen platen B which rotates in either direction around the Z axis and is central over an optical window in the platen providing transmitted illumination. The work tool (A) (Figure B.11) which rotates around the Y axis is mounted on a precision spindle and the specimen is centred under the work tool using the specimen stage X and Y micrometers. There are two types of tool, one for flat grinding and polishing and the other for dimpling. Flat grinding is used to level the specimen surface and employs a steel followed by a soft metal wheel such as brass (the same material removal mechanism as for tin lap (See B.5.1) and the coring tool (See B.6.1). The wheel is 3mm wide to cover the whole of the specimen area and has a diameter of 25mm. A grinding and polishing sequence using diamond pastes with an alcohol-based lubricant is followed by a fine polish using a soft polishing pad.

The dimpling tool shape is designed to wear a dimple in the centre of the specimen (Figure B.11). It has a smaller diameter of 16mm and width of 1.1mm, and the rim is chamfered at the edges to leave a rim thickness of 0.5mm and a more rounded shape. A grinding, polishing and fine polishing sequence is carried out as for the levelling process.

The precision spindle is bearing driven by a long pliable belt
Figure B.10  The VCR D500 Dimpler
HOW IT WORKS

Figure B.11 Schematic diagram of Dimpler
which negates the motor vibration from the wheel C and a further belt drive is connected to the electric constant torque motor D. Counterbalance weights $E_1$ and $E_2$ enable the whole lever arm to be balanced exactly at any motor speed selected. The oil dash pot F smooths out any motor vibrations transmitted through the body of the Dimpler. Loading weights can be selected in 5gm increments giving accurately controlled dimpling rates and a Z micrometer can be adjusted to terminate automatically for a desired thickness. An electronic timer can record the time elapsed before the Z terminator stops the dimpling process, or it can be used to stop the process at a pre-determined time. This allows the operator to continue other work.

A 3mm disc was glued on the circular optical window in the centre of the specimen platen with cyanoacrylate adhesive by using a mounting jig and a magnifier. The specimen surface must be coplanar and perpendicular to the work tools. For the flat grinding stage a steel wheel with a 6μm diamond slurry was used with a wheel speed of 40 on the machine arbitrary scale and a load of 40gm. By using the Z terminator to allow a known depth of material to be removed, together with the automatic timer which signalled the cut-out time, a grinding rate could be measured and was found to be around 1.2μm/min. Knowing this future grinding could be then carried out just by inputting a set time to remove a certain thickness. This avoids difficulties in using the Z terminator for grinding and polishing, where material debris builds up under the wheel surface, causing errors in the Z height measurement, and inefficient lapping. The 1μm polishing stage with the same weight and speed conditions gave material removal rates of around 0.08μm/min. For the final surface finish the soft polishing pad was used with a 1μm diamond slurry for a polishing time of up to three hours, and it produced a surface as shown in Figure B.12. The total apparent porosity is the same as for a 1μm tin lap polish of a 3mm disc but the surface scratches are eliminated. This advantage is counter-balanced by the inevitable surface relief.

From time to time it was necessary to true the 6 micron and 1 micron flattening wheels. This was done by placing silicon carbide wet paper on a specimen platen, using water as a lubricant, and applying a load of 100gm.
Figure B.12a  SEM micrographs of Dimpler 1μm diamond polished surface taken at a) 0°, 1,000x b) 60°, 3,5000x c) 60°, 9,000x
For the preparation of TEM specimens the same process was used as for the lapping stage. The steel dimpling wheel was first used with 6 micron slurry at the same conditions, giving material removal rates of around 1.3 microns/min. This also tended to be slowed down by the build up of material debris, which also caused difficulties in dimpling accurately to precise thicknesses using the 2 terminator. Dimpling was carried out to 6 microns higher than the intended final central specimen thickness of 25 microns to allow the 1 micron polishing stage to remove the damage from the 6 micron dimpling stage. Material removal rates were about 0.04 micron/min, but much slower if material debris was not cleaned from the wheel edge during dimpling. Central specimen thicknesses lower than 25 microns were not achievable as the specimens tended to crack. Finally the dimpled area was polished with a soft pad dimpling wheel with a 1 micron diamond slurry.